NINTH

HATFIELD MEMORIAL LECTURE

Fracture in Metals

By N. F. Mott

My aim in this lecture is to discuss three types of fracture in metals, ductile fracture, brittle fracture especially of ferrous materials, and fatigue. I shall discuss fracture in terms of the theory of dislocations and I will begin by outlining this theory and showing some small part of the experimental evidence on which it is now based.

DISLOCATIONS

WHEN G. I. Taylor[1] introduced the concept of a dislocation in 1934, his aim was to find a model which would explain the relatively low yield stress of metals and particularly of metallic single crystals. He suggested that metal crystals and crystalline grains normally contain dislocations of the type illustrated in Fig. 1 (taken from his paper), and that, when a stress is applied, shear takes place through the movement of these dislocations. This hypothesis has now a great deal of experimental support. The use of the bubble model[2] illustrates very well the forms that dislocations can take in a crystalline model and how slip takes place through their movement. Figure 2 shows a crystalline arrangement of bubbles containing a dislocation, and most of you will have seen the cinema film which shows its movement. In real materials, no technique has yet been devised for showing a dislocation in motion, but there is strong evidence both that they exist and that slip bands are formed through movement of a considerable number of them. The electron microscope is not yet powerful enough to photograph the positions of atoms in monatomic substances, but in platinum phthalocyanine this can be done. Figure 3 shows a photograph due to Menter[3] of a slab of this material some 10 molecules thick, showing some well-defined edge dislocations.

Very recently, Bond and Andrus[4] have used crossed nicols to photograph the stress field round a dislocation in a very nearly perfect crystal of silicon, using infra-red light to which this substance is transparent (Fig. 4). J. W. Mitchell[5] and his colleagues in Bristol have been able to make the dislocation lines in silver bromide crystals visible under the microscope by condensing photolytic silver on them, and have thus revealed both the arrangements of dislocations within an annealed material, and have shown that there is a large concentration of dislocations along the slip lines.[6] These photographs have been reprinted in many articles[7, 8, 9] and will not be reproduced here.

One thing we do not know about dislocations is the stress necessary to make one move in an otherwise perfect crystal. This is known in the literature as the Peierls-Nabarro stress. In metals having the close-packed hexagonal or cubic structures, all the evidence suggests that it is very small, perhaps zero except at very low temperatures. In body-centred metals (e.g. iron), the position is not so clear; it is possible that at low temperatures it may be quite large. In materials with the diamond structure, diamond itself, silicon, and germanium (which are ductile if at all only at high temperatures), it is clear that the Peierls-Nabarro stress must be large at

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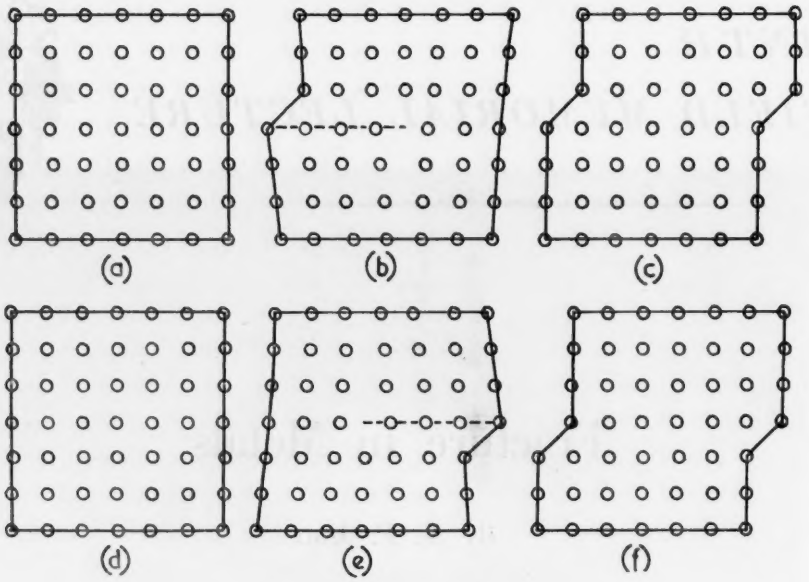


a, *b*, *c*, Positive dislocation *d*, *e*, *f*, Negative dislocation

Fig. 1—Dislocation in a crystalline material (*G. I. Taylor*[1])

low temperatures and fall off at high temperatures. This dependence on structure is perhaps not unexpected, since the movement of a dislocation in the diamond structure involves the breaking and reforming of bonds; however, no real calculation of the stress starting from known interatomic forces has yet proved possible.

What is clear is that impurities, particularly interstitial impurities such as carbon or nitrogen in iron, have a strong tendency to segregate on the dislocation lines, producing a yield point and, as we shall see later, under certain circumstances embrittlement. The idea of this segregation and its effect on the yield point is due to Cottrell[10, 11, 12] and a whole series of papers is based on it. The idea is that the impurities lock the dislocation so that the stress S required to move it away from its line of impurity atoms is greatly increased. Both theoretical analysis (Cottrell and Bilby[11]) and observations of the upper yield point (supposed to be identical with it) (McAdam and Mebs[13]) show that S is strongly dependent on the temperature, dropping by a factor

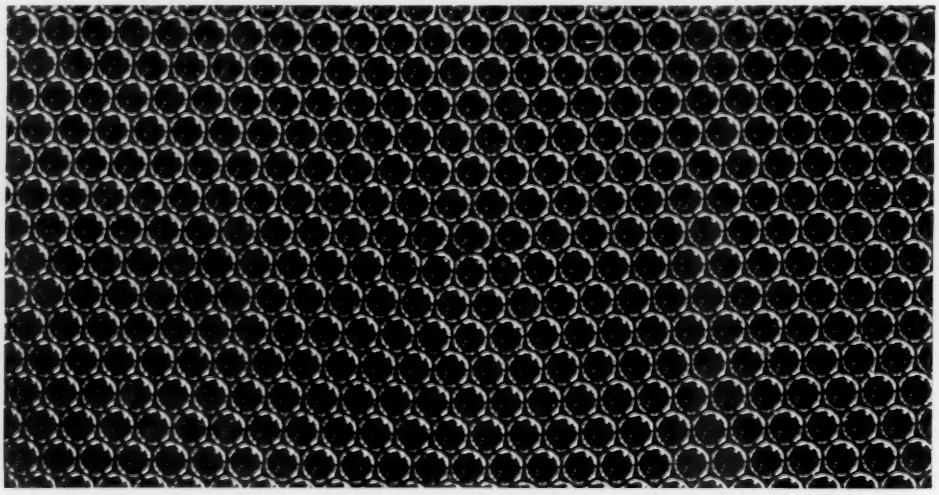
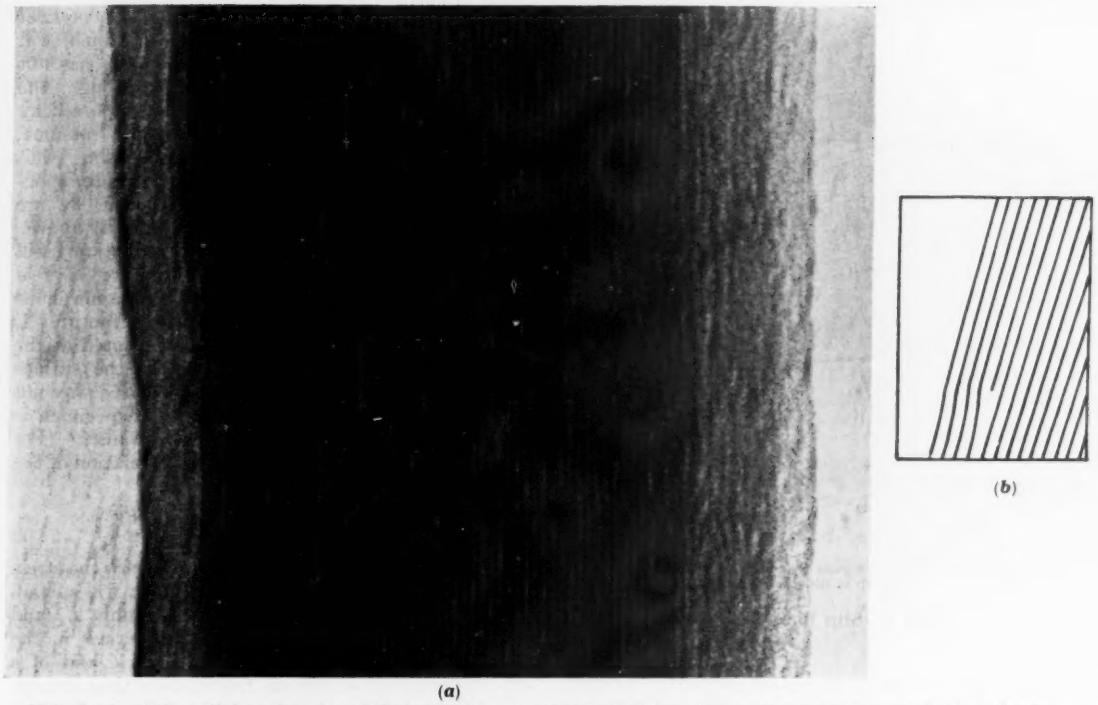


Fig. 2—Crystalline raft of atoms containing a dislocation (*Bragg and Nye*,[2] *Plate 10, Fig. 6a*)



(*a*)

Fig. 3—(*a*) Edge dislocation in phthalocyanine, photographed by direct transmission with the electron microscope through a crystal of thickness about 200 Å (*Menter*[3]) (*b*) Dislocation in phthalocyanine

4·1 between the temperatures −196° C and 20° C.

When once yield begins in a metal, one of the oldest observations of metallography is that slip is not uniformly distributed through the material but partly,*

* c.f. observations of Wilsdorf[14] *et al.* on fine slip.

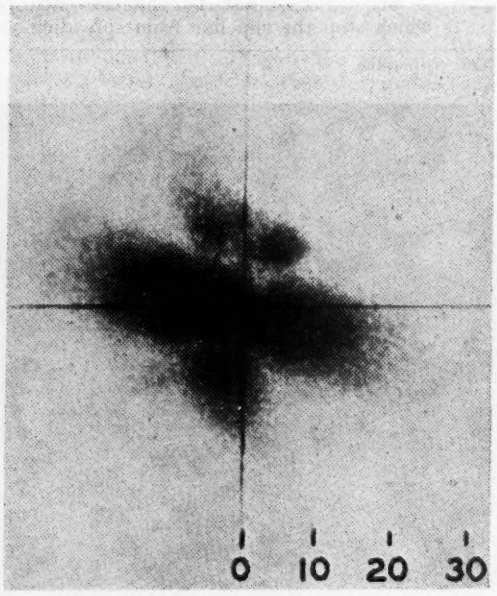
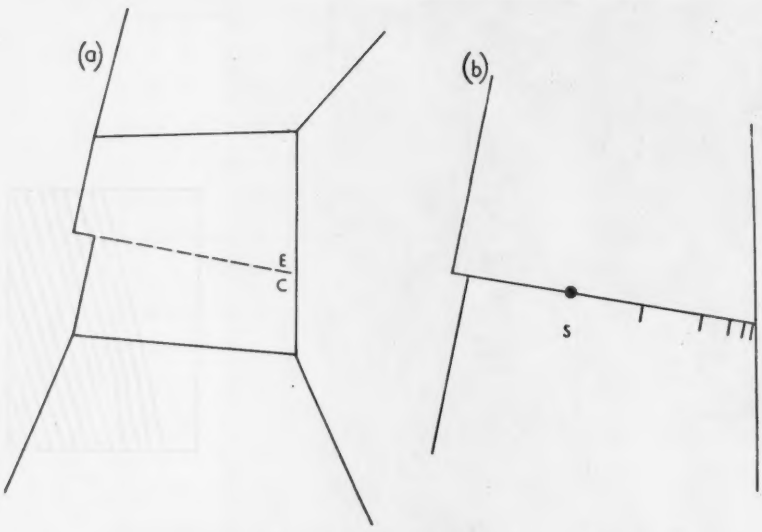


Fig. 4—Dislocation in silicon photographed with infra-red light by crossed nicols (*Bond and Andrus*[4])

if not wholly, localized on certain active slip planes some microns apart on which slip may occur through several thousand atomic distances. Since we believe that slip occurs through the motion of a dislocation along a slip plane, and since the passage of a single dislocation produces slip through one atomic distance, the assumption was early made, particularly by Masing[15] and his colleagues in Göttingen, that there must exist in the material *sources* of dislocations which could produce dislocations one after the other when the material is stressed. An extremely promising hypothesis of the nature of the sources was given by Frank and Read,[16] which, though no experimental proof of it has yet been devised, is fairly generally accepted. They show that a certain arrangement of dislocations, when stressed, is capable of producing new ones. This arrangement is called a ' Frank-Read source.' In this lecture I shall at any rate assume that sources of some kind exist, and that sources as well as single dislocations are capable of being locked by impurities.

BRITTLE AND DUCTILE FRACTURE

To understand fracture we have next to ask what will happen when an active slip plane terminates on a grain boundary as illustrated schematically in Fig. 5*a*. Even without any consideration of dislocation it is clear that the region C must be highly compressed, and the region E expanded. In terms of dislocations, the theoretical analysis of Frank, Eshelby, and Nabarro[17] leads us to expect the arrangement of dislocations shown in Fig. 5*b*, a group of dislocations being ' piled up ' against the



(a) Compressed and expanded regions E and C where the slip line meets the grain boundary; (b) Piled-up group of dislocations, originating from the source S.

Fig. 5—Slip lines in a polycrystalline material

barrier which the grain boundary presents. Fortunately, piled-up groups arranged exactly as the theory suggests have been observed by Jacquet[18] in brass, the position of each dislocation being shown by an etch pit formed under the action of a suitable reagent (Fig. 6).

Theoretical analysis shows that an active slip plane containing dislocations behaves like a freely-slipping crack, so that it will raise the stress much as a crack will. The amount it will raise it depends on the length L of the crack. When the applied shear stress S exceeds a critical value of order*

$$S = G\sqrt{(b/L)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

(where G is the elastic shear modulus and b the interatomic distance), the material at the end of the slip line must give way. There are three modes by which it can give way:

(i) It can crack, as illustrated in Fig. 7

(ii) It can slip, the stress being great enough locally to shift one plane of atoms over the other without the prior existence of dislocations

(iii) It can slip, making use of dislocations generated at sources in the vicinity.

* A derivation of this formula is given in the Appendix. It could be written in the form

$$S = \text{const.}\ \sqrt{(G\gamma/L)} \ldots\ldots (1.1)$$

where γ is the surface energy, but for actual metals γ is closely proportional to Gb.

A convincing theoretical analysis to show which will occur first, (i) or (ii), has not yet been given though the problem has been discussed by Stroh.[19] We shall assume, following other workers, that cracking occurs before spontaneous slip, so that we are limited to (i) and (iii) above.

With this introduction I will turn to the fracture of real materials. For amorphous materials (glass) fracture is believed to be initiated at the Griffith cracks at the surface which are formed in a way not understood as the specimen is cooled (cf. Orowan[20]). For Griffith cracks a relation of the type† (1)

$$S = \text{const.}\ \sqrt{(E\gamma/\lambda)} = \text{const.}\ E\sqrt{(b/\lambda)} \quad \ldots\ldots (2)$$

also exists between S, the stress at which the crack will spread, the Young's modulus E, and the depth of the crack λ; the lengths of a crack and of a slip line required to initiate fracture are thus similar.

I do not believe that brittle or cleavage fracture in metals or other crystalline materials is due to Griffith cracks. It is more likely that it is normally initiated by slip in the way that I have described. I will now attempt to describe the conditions under which such fracture can occur in a polycrystalline metal such as iron or steel.

The first condition for fracture due to piled-up groups of dislocations must be the existence of barriers which stop the slip line from spreading, so

† cf. Appendix.

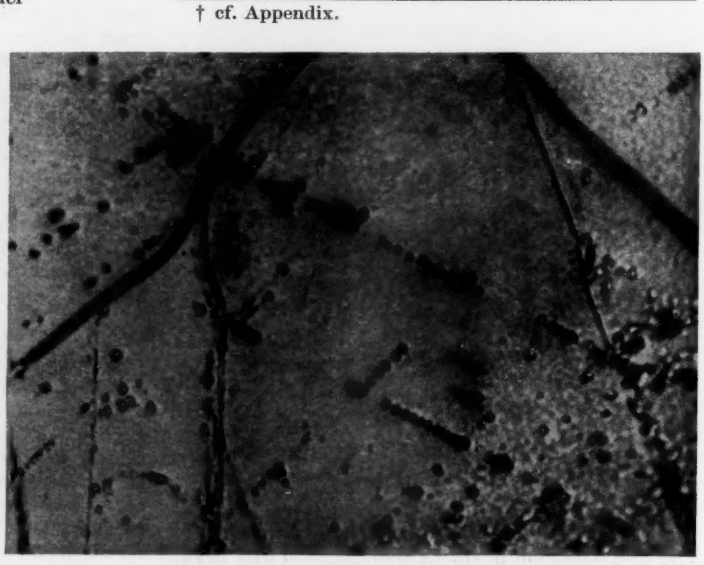


Fig. 6—Piled-up groups of dislocations in polycrystalline brass (*Jacquet*[18])

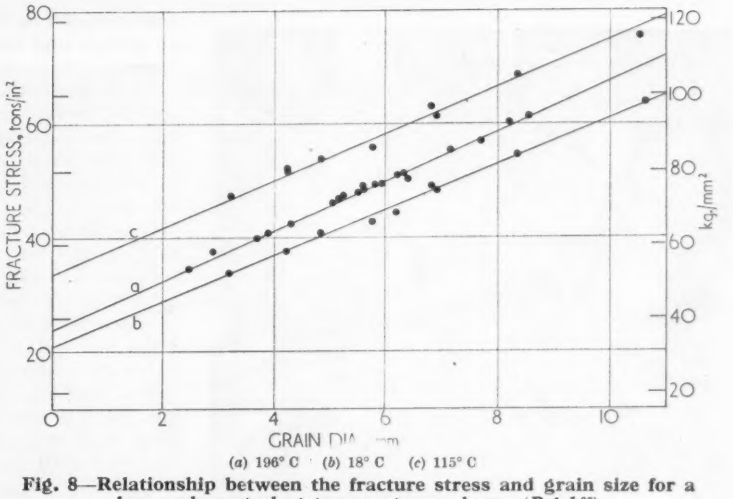
that the postulated high tensile stresses will build up there. We believe these to be grain boundaries. Ductile (cup-and-cone) fracture does not occur in single crystals, which normally show 100% ductility and break by pulling down to a knife edge. Brittle fracture of single crystals of hexagonal metals and of iron at low temperatures is certainly observed (cf. Allen, Hopkins, and McLennan[21]); no satisfactory theory has been given of this process and we shall confine this discussion to polycrystalline metals.

In polycrystalline metals then, the conditions for fracture will be as follows. Brittle fracture will result if a slip line crosses a grain of diameter L, if the *shear* stress is greater than the critical stress given by (1), if the crack which it initiates is not damped out by plastic flow round its apex, and if the *tensile* stress is great enough to make it spread. The mechanism for this damping will be discussed below. If the crack is damped out, then we shall expect ductile fracture when eventually a large number of these cracks is formed, and they join together (e.g. *see* Mott,[7] Petch[23]). For both processes, brittle and ductile fracture, however, we should expect the same relationship between the grain size L and the fracture stress S, viz.

$$S - S_0 = c.\ G\sqrt{(b/L)} \quad \ldots\ldots\ldots\ldots\ldots(3)$$

Here S_0 is the frictional force which one assumes to oppose the movement of a dislocation in a cold-worked metal, due to its interaction with all the other dislocations.

Figure 8, due to Petch,[23] shows the relation between the fracture stress S of low-carbon steel and the grain size L, at various temperatures. At −196° C the fracture is brittle without apparent prior deformation, at the other temperatures a normal cup-and-cone fracture is obtained after some 60% reduction in area. It will be seen that the curves follow closely the predicted form. The value of the constant c in equation (3) can be calculated, depending as it



(*a*) 196° C (*b*) 18° C (*c*) 115° C

Fig. 8—Relationship between the fracture stress and grain size for a low-carbon steel at temperatures shown (*Petch*[22])

does only on the shear modulus, surface energy, and atomic constants. Values are given by Stroh. Stroh expresses the equation (3) in the form:

$$(S - S_0)L^{\frac{1}{2}} = 4\{b\gamma G/\pi(1-\nu)\}^{\frac{1}{2}} \quad \ldots\ldots\ldots\ldots\ldots(4)$$

where γ is the surface energy and ν Poisson's ratio; the observed and calculated values of the quantity are on the right-hand side (in dyn/cm × 10^{-8}) in Table I

Here the surface energy is taken from Taylor[28] and G is the bulk modulus of the polycrystalline material. It is noteworthy that for zinc Greenwood and Quarrell find that S_0 is negligibly small; why this should be so is not clear. The agreement between theory and experiment is good enough to suggest strongly that the theory is on the right lines for the calculation of c.

Our next problem is to ask whether or not the crack formed at the end of a slip line will spread. We must first ask to what length the crack will extend under the action of the shear stress only. This has been calculated by Stroh[19] (formula (2) of his paper); the answer that he obtains is:

$$L\ (LS^2/bG^2) \quad \ldots\ldots\ldots\ldots\ldots(5)$$

If S satisfies (1) this is about the length L of the slip line itself. The crack will not grow at all unless, according to the Griffith criterion (2), a *tensile* stress of comparable order of magnitude is present.

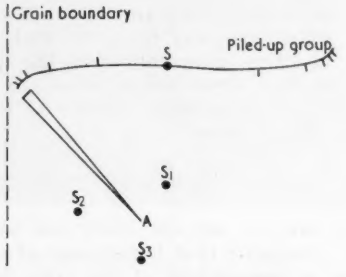


Fig. 7—Crack at the end of a slip line of length L, together with surrounding sources of dislocations

Table I

OBSERVED AND CALCULATED VALUES OF CONSTANT *c*

Element	Calculated according to (4)	Experiment	Authority
Fe	2·4	2·0	Petch[23, 24]
Zn	1·1	1·2	Greenwood and Quarrell[25]
Mg	0·89	0·69	Hauser, Landon, and Dorn[26]
Mo	3·4	3·9	Bechtold[27]

Fig. 9—Centre of a fatigue-crack in polycrystalline copper (*Thompson, Wadsworth, and Louat*[36])

Assuming, however, that such a stress is present, we have to ask whether it will spread, or whether it will be damped out by movement of dislocations generated from the surrounding sources marked S in Fig. 7. If it spreads, a crack will be propagated with speed near to that of sound; if not, then the crack can only extend to a distance comparable with l, the distance between sources in the material, say a micron. Now the stress due to the crack acting at any one of these sources will be of order

$$S\sqrt{(L/l)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(6)$$

Since for fracture, (1) is satisfied, this may be written

$$G\sqrt{(b/l)} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(7)$$

Now the theory of the Frank-Read source suggests that, in a material containing a uniform network of dislocations, a source will act if a stress is applied which is greater than Gb/l, and this is much less than (7); it follows that, *unless the sources are locked*, e.g. by impurities, cracks cannot spread. This corresponds to the observed fact that in face-centred cubic metals, where locking of dislocations is weak and yield points are absent, ductile fracture occurs, presumably by the formation of a large number of microcracks of this sort which run into each other (Mott[7]).

The observations of Petch[23] (Fig. 8) on the ductile fracture of iron, showing that a relationship of the type (3) exists here as well, support the theory that it is due to groups piled-up against grain boundaries. We have already remarked that ductile fracture does not occur in single crystals, and the work of Stroh[29] shows that the 'sessile' dislocations of Cottrell[30] and Lomer[31] (e.g. *see* Mott[4]) do not provide strong enough barriers for this type of fracture. It should, however, be mentioned that other theories of ductile fracture have recently been proposed (Mason[32]) which depend on the formation of vacancies by piled-up groups and are not connected with grain boundaries.

In ferrous materials, unless very pure, the sources will be locked by carbon, nitrogen, or hydrogen, and there remains the possibility that, even in the purest materials, there may be a locking effect due to the Peierls-Nabarro stress. We then have to ask whether, in the time available, the source will act and generate dislocations. This will depend on the temperature, since we have seen that the stress required to cause a dislocation to break away from its locking atmosphere is very sensitive to temperature. We thus have to ask whether the stress (7), with l of order 1μ, will or will not be sufficient to tear the dislocation away in the time available. The time will be that required for n dislocations to traverse the slip line; if they move with a velocity not too far from that of sound, v, this should be about nL/v, or if L is 0·01 cm and n is 1000, say 10^{-4} s. Stroh[22] has made an estimate of the temperature below which this stress would be sufficient, using the theory of Cottrell and Bilby[11] of the thermally activated process by which dislocations break away from carbon atmospheres; he obtains 300° K. The estimate is clearly subject to several uncertainties, but is of the right order for the brittle-ductile transition in steel. An interesting experiment would be to measure the transition temperatures in specimens of the same geometrical forms in pure iron with yield points introduced by carbon, nitrogen, and hydrogen respectively; theoretical calculation of the *relative* transition temperatures could be made.

Stroh also considers the experiments of Robertson[33] in which a crack started from a saw cut is propagated through a temperature gradient and the temperature where it is arrested is observed. This temperature will be higher than that at which a crack, formed by the shear stress as outlined in Fig. 7, will begin to propagate under the influence of the tensile stress. The reason is that the surrounding sources will be subject to the high stress $S\sqrt{(\lambda/l)}$ (where λ is the length of the crack) only for a much shorter time of order v/l or 10^{-10} s, and also that a considerable generation of dislocations will be necessary to damp out the crack. How much has not been worked out.

To conclude this section we may mention the analysis of the lower yield point given by Cottrell and Bilby.[11] According to these authors this is the stress at which stress round the piled-up group (not the crack) will cause nearby sources to generate dislocations. The stress acting on such sources will be given by a formula of type (6); we have to ask whether, in a time comparable with the time of the experiment, this stress will be equal to that (the upper yield point) required to operate the source. In fact the theory gives

$$S_L = \sqrt{(l/L)}S_Y$$

where S_L and S_Y are the lower and upper yield stresses. Assuming that l, the scale of the microstructure, is independent of the grain size L, we obtain the constancy of $S\sqrt{L}$ observed by Hall[34] and Petch[24]. Stroh[22] has an ingenious argument

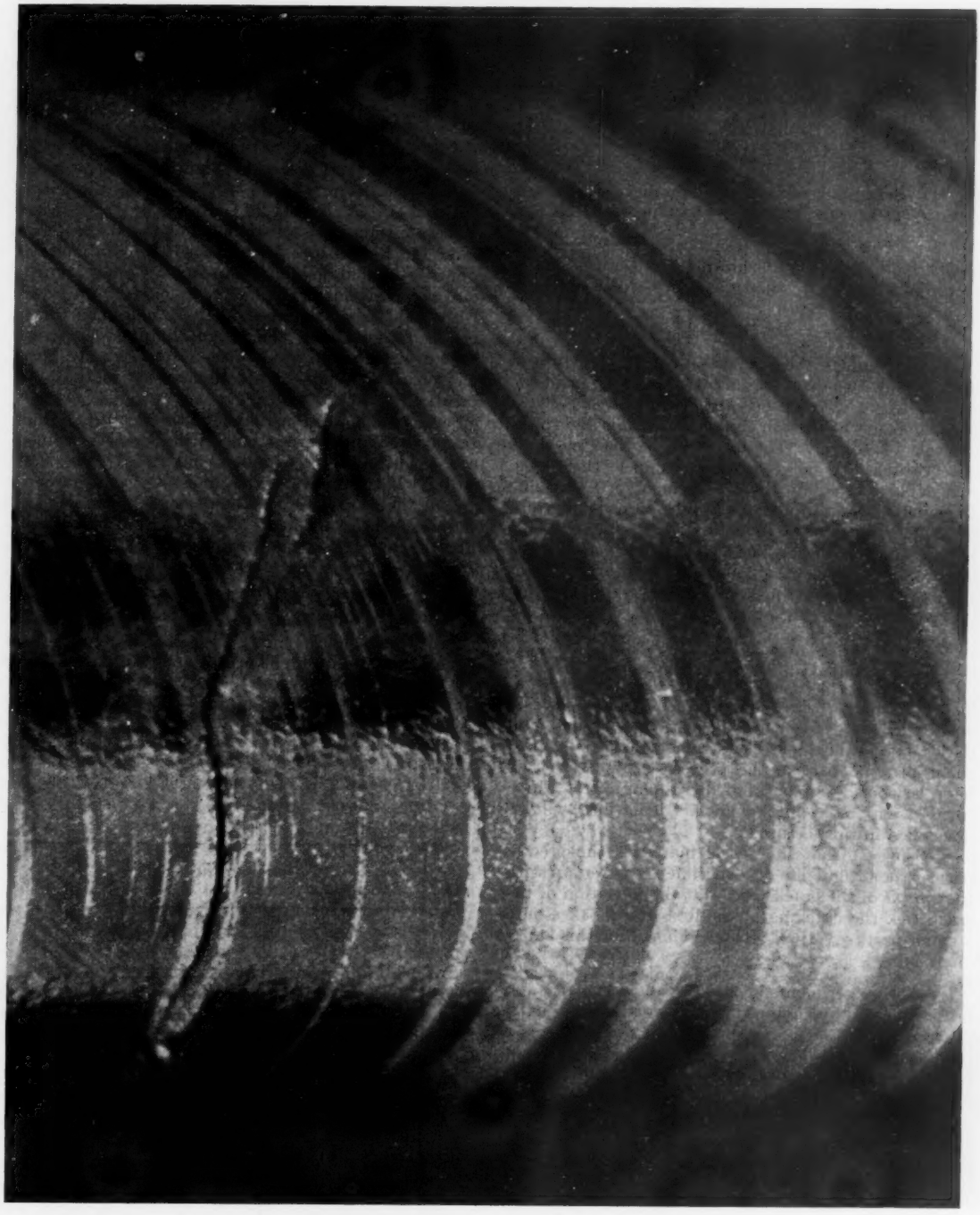


Fig. 10—Fatigue-crack in a single crystal of copper (*Thompson*, unpublished). Note the circumferential glide pockets and that much of the crack lies on one of these × 60

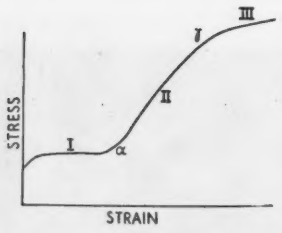


Fig. 11—Schematic work-hardening curve of a single crystal of a face-centred cubic metal

about this constancy of l, showing that sources nearer to the piled-up group than $l_{crit} = Gb/S_T$ cannot act.

FATIGUE

We turn now to the problem of fatigue. Figures 9 and 10 show two examples of fatigue cracks, from the researches of Thompson and Wadsworth[35, 36] at the University of Bristol, on polycrystalline and single crystals of copper. The first shows that in these observations in polycrystalline copper the crack invariably started by the enlargement of a slip line in a single grain into a crack, which spreads out into neighbouring grains at an angle of approximately 45°. Figure 10 shows the start of a fatigue crack in a single crystal.

For polycrystalline materials, then, we may divide the fatigue process into

(i) The initiation of the crack in a single grain
(ii) Its gradual spread throughout the material.

The fact that a very similar process takes place in single crystals leads us to believe that grain boundaries and groups of dislocations piled up against them play no part in the process (i), and that we have to consider some kind of rubbing effect which turns a slip band into a crack. This idea will be developed in this lecture.

We suggest that in ductile materials the greater part of the fatigue life of a specimen is taken up with the *spread* of the crack, and that the initiation of the crack (i.e. the cracking of the first grain) takes only a small part, say 10%. Our evidence for this statement is:

(*a*) The experiments of Dolan and Sinclair[37] on brass, repeated by Thompson, Wadsworth, and Louat[36] on copper, that frequent annealing does not increase the fatigue life. The damage early on thus cannot be removed by annealing and must be of the nature of the crack.

(*b*) The analysis of Head,[38] which suggests that the length λ of a fatigue crack grows according to the formula

$$\lambda^{-\frac{1}{2}} = f(S)\{n_S - n\}$$

where $f(S)$ is a function of the stress, n the number of cycles, and n_S the number of cycles at which fracture ($\lambda = \infty$) occurs for the stress considered. Head compares this formula with experimental data due to Moore,[39] de Forest,[49] and Bennett[41] and finds that, on extrapolating back to the grain size, one accounts for the greater part of the life.

(*c*) The observations of Thompson, Wadsworth, and Louat[36] that if the surface is removed after a considerable amount of fatigue, the (hardened) material remaining has about the same fatigue life as the virgin specimen.

It follows then that the effect (Gough and Sopwith[42]) of oxygen on the fatigue life is mainly on stage (2), the spread of the crack. I known of no evidence which would show whether oxygen affects the initiation of the crack, neither do I know of any hypothesis on how oxygen affects the spread of the crack. The work of Rosenberg (unpublished) on fatigue of copper at liquid helium temperatures show clearly that oxygen is not necessary.

I would hazard the guess that for materials which show a fatigue limit, the fatigue limit is the stress below which this initiation does not occur and above this the fatigue life is determined by the rate of spread of a crack.

For most of the remainder of this lecture I shall consider the initial process in which a crack is formed in a single grain. To understand this I must first outline what is known of the process of work-hardening in cubic metals. When single crystals are extended, the work-hardening can be divided into three main stages, shown in Fig. 11; these are (cf. Friedel[43] for a summary of the data):

Stage I, known as 'easy glide' (*Andrade and Henderson*[44]). This occurs in single crystals of cubic metals when glide is limited to one system of slip planes; it is absent in polycrystalline materials. Fine slip lines cross the whole crystal and hardening is slow because few dislocations are stored in the crystal.

Stage II, the region of rapid hardening. This begins when two or more slip systems are brought

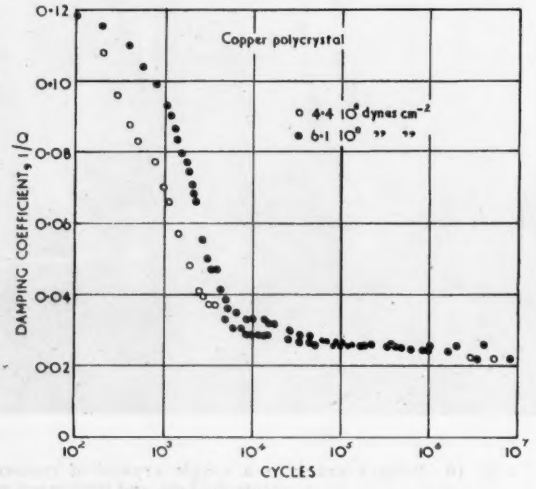
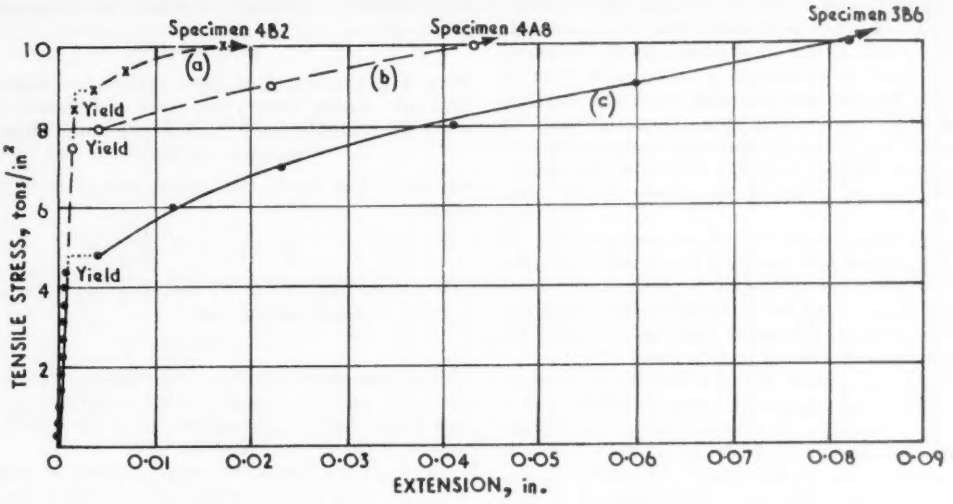


Fig. 12—Damping of polycrystalline copper in high speed fatigue test (*Wadsworth*[54])



(*a*) After cyclic stressing at ± 9 tons/in² (*b*) After cyclic stressing at ± 8 tons/in² (*c*) In initial condition

Fig. 13—Stress-extension curves up to 10 tons/in² in specimens (*Wood and Thorpe*[47])

into play. In this region, if the surface is electropolished at some point on the stress/strain curve (such as *A*) and then strained further, *short* slip lines are formed (Blewitt, Coltman, and Redman[45]). These are believed to occur because, when two dislocations on different slip systems run into each other, they form in certain circumstances a locked or sessile dislocation (theory of Cottrell[30] and Lomer[31]). Since the dislocations cannot move far, many more of them have to be formed to produce a given strain, and the material hardens rapidly. A detailed theory of this kind of hardening has been given by Friedel,[43] which has proved a considerable help in interpreting it, though our understanding of it is by no means complete.

In Stage III only, do the familiar long, coarse slip lines occur. According to the ideas of Friedel,[43] of Seeger,[46] of Stroh,[29] and of the present author,[47] these form because certain of the Cottrell-Lomer barriers break down under the combined influence of stress and temperature, so that the dislocations from the source concerned spread across the crystal. Since now the dislocations can once more move a long way, as in stage I, hardening will be slow.

Turning now to the behaviour under reversed stress, the recent work of Paterson[48] shows that easy glide occurs under repeated reversals in single crystals when the orientation is favourable, as in unidirectional stress. One knows too, from much evidence, e.g. Wood and Thorpe,[49] that hardening occurs in cyclic stressing much as in unidirectional stressing, the chief difference being the absence of the cell formation revealed by X-ray diffraction which presumably is dependent on the gross bending of the grains. I would hazard a guess, then, supported to some extent by theoretical considerations (Mott[47]), that the three stages of hardening exist under cyclic straining, e.g. with a constant strain at each cycle. Experiments such as those of Blewitt *et al.*,[45] in which the surface is polished at various stages of the test and the slip-lines observed after further testing, would be of considerable interest.

If this is so, observations such as those of Fig. 9 suggest that in polycrystalline materials, fatigue will occur when slip lines of the stage III type cross a grain; the slip lines in stage II will be too short. As a further hypothesis I may suggest that any slip line is a source of weakness, liable to develop into a crack, and the longer the slip line the longer (and more dangerous) the crack. We have to ask, then, under what conditions does a slip line develop into a crack.

We have no clear answer to this question and I can only speculate about it. I have already said that I do not think that it can be anything to do with the formation of piled-up groups of dislocations, since the effect occurs in single crystals. It has been widely suggested that the effect is due to the formation of vacant lattice sites by moving dislocations. It is true that there is much evidence (*see* Broom[50] for a summary) that moving dislocations do produce point defects, so that if dislocations move backwards and forwards in slip bands, we have the possibility that the lattice may be completely disordered here, and it will expand and then contract locally when it recrystallizes. Such a process may lead to cracking. Another possibility is that fracture is normally caused by the extrusion process observed in aluminium alloys by Forsyth[51]; to explain this one would have to understand why a moving dislocation *softened* the material in its immediate neighbourhood; for this more than one explanation is possible (Mott[47]).

I think that in the present state of knowledge our

most profitable course will be to consider whether, in a fatigue test, there is evidence that dislocations do move backwards in slip lines during cyclic stressing. Though Louat[52] has obtained a photograph showing that a slip line reverses entirely when the stress is reversed, the observation has been queried by Thompson[53] and certainly cannot happen to most of the slip lines during fatigue. If it did, the damping would be an order of magnitude higher than observed.

To show that this is so, let us consider Fig. 12 which reproduced the damping coefficient $1/Q$ after varying numbers of cycles of a specimen of polycrystalline copper used in the experiments of Thompson, Wadsworth, and Louat[36] mentioned above; the curve is taken from Wadsworth's thesis.[54]

The initial high value of $1/Q$ is due to plastic flow; when hardening is complete Q remains constant over a very large number of cycles. One may suppose that, since no hardening is going on, this is due to the movement backwards and forwards of dislocations in a reversible way. The question is, how many move.

Figure 2 of the paper by Wood and Thorpe already quoted is reproduced here as Fig. 13. It shows that, in these tests, cyclic stressing of a brass specimen hardened it about the same amount as 10% unidirectional strain. We have no similar data for Wadsworth's copper; however, let us assume that the values are similar. Then, if our hypothesis is correct that cyclic hardening and unidirectional hardening are essentially the same, we may assume in either case that per unit area in the crystal, n dislocations have moved a distance L when the strain nbL is given by

$$nbL \sim 0{\cdot}1$$

Now if a fraction f of these moves backwards and forwards during each cycle over a distance L, the work dissipated in each cycle is per unit volume

$$2SLfnb = 0{\cdot}2Sf$$

On the other hand, the stored energy is $S^2/2G$, so that

$$\frac{1}{Q} = \frac{0{\cdot}2Sf}{S^2/2G} = \frac{0{\cdot}4fG}{S}$$

Since according to Fig. 13 the observed value of $1/Q$ is 0·02, it would follow that

$$f \sim 0{\cdot}05S/G$$

The applied stress in these experiments was 5×10^8 dyn/cm^2, and for copper we have taken c_{44}, given by Boas and Mackenzie[55] as $0{\cdot}75 \times 10^{12}$. We find that f is of the order 10^{-4}.

On most slip bands, the step height is of order 1μ, corresponding to the passage of somewhat less than 10^4 dislocations. Thus if the dislocations are moving backwards and forwards on the slip lines, certainly their number cannot be more than one or two on each line. Thus it looks as if the initial fatigue crack was produced by the motion backwards and forwards of one or two dislocations, say 10^5 times.

We are still far from understanding how the slip lines form regions of weakness under cyclic stressing; however it seems established that they do so act, and the next step in a systematic investigation of the phenomen is to find out how they act.

APPENDIX

Stroh[19] gives the following formula for the stress S necessary to form a crack at the end of a slip line

$$S \sim 12\gamma/nb \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (8)$$

Here γ is the surface energy, b the Burgers vector, and n the number of dislocations in the piled-up group, so that, according to the theory of Eshelby, Frank, and Nabarro,[17] the stress at the leading dislocation is nS. Stroh envisages that a crack will be formed when the stress is large enough for two or more dislocations to be forced together as in Fig. 14. The surface energy for all metals is, according to Stroh, using Taylor's[28] experimental data, related to the shear modulus by an empirical relation of the type

$$8\gamma \sim Gb,$$

so (8) becomes

$$S/G \sim 1{\cdot}5n.$$

Now n, the number of piled-up dislocations in the group, depends on the length $2L$ of the slip line (L is the slip distance for dislocations of a given sign), and is given by the theory of Eshelby *et al.*[17] by

$$n = \pi LS/Gb$$

whence

$$S/G = (b/L)^{\frac{1}{2}} \quad \ldots\ldots\ldots\ldots\ldots\ldots (9)$$

We may compare this with Griffith's formula for the propagation of a crack by a *tensile* stress S

$$S = \{2\gamma E/\pi L(1-\nu^2)\}^{\frac{1}{2}}$$

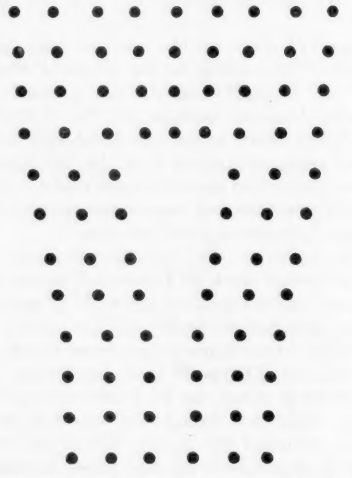


Fig. 14—Dislocation with Burgers vector $3b$, showing an incipient crack (*Stroh*[19], Fig. 1)

(where E is Young's modulus and ν *Poisson's* ratio), which, when we substitute for the surface energy γ in terms of G, gives

$$S/\sqrt{(EG)} = \{4\pi(1-\nu^2)\}^{\frac{1}{2}} \sqrt{(b/L)} \quad \ldots\ldots\ldots\ldots (10)$$

a very similar formula though with a *smaller* numerical coefficient in the right-hand side. Either formula (9) or (10) may be interpreted as follows: near the apex of a crack the stress falls off with distance r from the apex as

$$S(L/r)^{\frac{1}{2}}$$

The condition that a crack should be initiated is that at one atomic distance ($r = b$) this should be of order G (or $\sqrt{(G\gamma b)}$).

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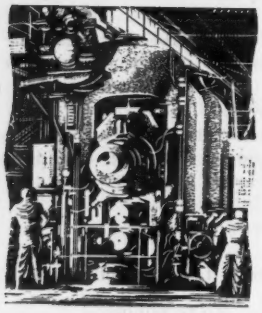

PAPERS AND REPORTS ON RESEARCH AND PRACTICE

Metallurgy · Plant Operation · Research

The Alloying of Tin and Iron during Flow-brightening of Electro-tinplate

By C. J. Thwaites, B.Sc., A.R.S.M., A.I.M.

SYNOPSIS

The rate of formation of the intermetallic compound $FeSn_2$ during the flow-brightening of electrolytic tinplate has been studied, using laboratory equipment which simulates commercial tinplate practice, and was found to be initially extremely rapid. The effect of different heating conditions was observed and it was shown that considerable alloying occurs at temperatures below the melting point of tin. Metallographic examination of the $FeSn_2$ layer showed the effect of alloy thickness on the structure and the mode of melting of the tin during flow-brightening was observed by examination of the surface of the tin coating. Reflectivity measurements showed the effect of flow-brightening temperature and of tin coating-weight on the brightness of the tinplate.

1177

ELECTROLYTIC TINPLATE is manufactured by passing a continuous band of mild-steel strip at high speed through a series of units in which it is cleaned, pickled, and electroplated with tin.[1] The electro-tinned strip is then normally passed into a heating tower where the tin coating is momentarily fused and immediately quenched in water. This ' flow-brightening ' operation causes alloying between the tin and the steel base and also imparts to the tinplate a surface of high reflectivity and good solderability.

There are few published data on the rate of alloy formation during the flow-brightening of electro-tinplate, the work of other investigators being concerned mainly with the alloying occurring when steel is immersed in a bath of molten tin[2] and with the metallography of the tin–iron alloy layer.[3–6] It thus seemed desirable to study this phenomenon fundamentally, and the present paper describes the examination of the effect of varying time/temperature relationships on the rate of formation and on the metallography of the alloy layer.

EXPERIMENTAL PROCEDURE

Before the study could be attempted, it was a necessary preliminary to find a method for measuring the amount of alloy on relatively small samples with a satisfactory degree of accuracy. The coulometric method developed by Kunze and Willey[7] appeared to satisfy the requirements and the results, published elsewhere,[8] of extensive check tests against standard techniques confirmed that determinations could be made with the necessary accuracy and with less experimental difficulty than would have been experienced with other techniques.

Specimens of tinplate steel base were electroplated with various amounts of tin in a circulating electrolyte cell which simulated plating conditions in an industrial tinplate line. Flow-brightening of the plated samples was carried out by electric-resistance heating, which was deemed to be the most convenient method for laboratory experiments and is, in any case, most commonly used in industry. The total time of heating was varied, and the maximum temperature reached by the specimen before quenching and the corresponding amount of alloy formed were measured. The effects of different rates of heating and of heating to a given temperature followed by holding at that level for various times were also studied. The rate of alloying occurring below the melting point of tin[7] was examined by immersing samples for various times in an oil bath held at given temperatures. Many specimens were examined under the microscope to observe the mode of formation of the alloy layer and its

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Table I
AVERAGE COMPOSITION OF STEEL BASE

C, %	S, %	P, %	Si, %	Mn, %	Cu, %
0·085	0·059	0·040	0·001	0·20	0·050

changes in appearance with thickness and temperature. The microscopic appearance of the surface of the tin coating was also examined in detail and tests were made to find the effect of flow-brightening conditions on the reflectivity.

DESCRIPTION OF APPARATUS

The Circulating Electrolyte Plating Cell

The circulating electrolyte plating cell has been described elsewhere[9] and only a brief account will be given here.

Essentially, the cell comprises a shallow rectangular box, the bottom of which is a block of pure tin serving as the anode, the top being closed by a steel specimen forming the cathode, backed by a tinned-copper plate. A closed circuit of pipework connects this unit in series with a pump and a reservoir tank containing the electrolyte which, in this instance, is of the acid stannous phenolsulphonate type. On starting the pump the solution is circulated and passes between the anode and cathode at a velocity of 600 ft/min.

The steel samples to be plated (11·3 in. × 3·9 in. × 0·01 in.), were cut from a single batch of British cold-reduced tinplate base, the composition of which is shown in Table I.

Each sample was initially treated cathodically for 3 min., at a current density of 50 A/ft² and a temperature of 80–90° C in an alkaline cleaning bath. Pickling was by immersing for 2 s in inhibited 2% sulphuric acid at 85° C. Plating was carried out at a current density of 100 A/ft², which resulted in a rate of deposition of tin of 1 oz per basis box (0·7 g/m²) per second or a coating of approximately 4μin. thickness per second.

The Flow-Brightening Units

Two flow-brightening units were used for the experimental work, one with a specimen size of 10½ in. × 3½ in. and a smaller one for a specimen 6 in. × 2¼ in. (*see* Fig. 1). The large apparatus, described previously,[9] provides a single high rate of heating of the specimen and was designed in the first instance for laboratory production, under standardized conditions, of large quantities of samples for another research. In the second apparatus a sample can be heated for a known time at varying rates and kept at a constant temperature for a pre-determined time.

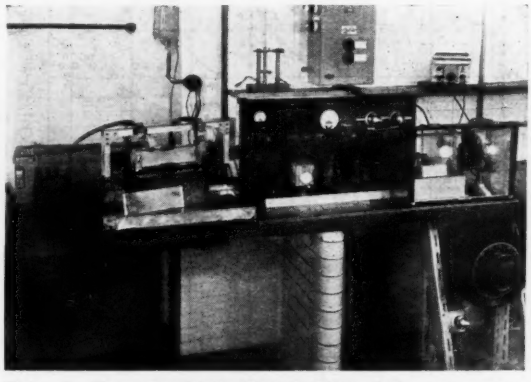


Fig. 1—Experimental flow-brightening apparatus. Left: large unit. Centre: control panel and timing equipment. Right: small unit in constant temperature box

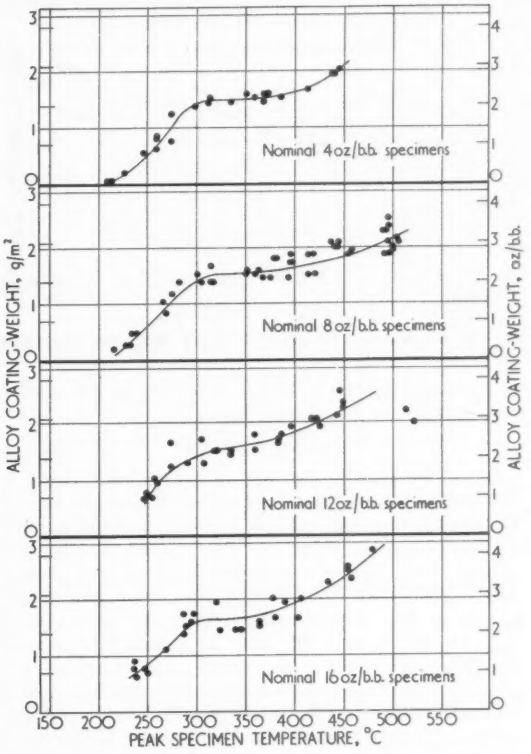

Nominal 4 oz/b.b. specimens
Nominal 8 oz/b.b. specimens
Nominal 12 oz/b.b. specimens
Nominal 16 oz/b.b. specimens
ALLOY COATING-WEIGHT, g/m²
ALLOY COATING-WEIGHT, oz/b.b.
PEAK SPECIMEN TEMPERATURE, °C
150 200 250 300 350 400 450 500 550

Fig. 2—Relationship between amount of alloy and peak specimen temperature for four total tin coating weights

Each apparatus consists of a framework of two heavy copper blocks slotted to accommodate the specimen and held rigidly a fixed distance apart. Current is supplied to the specimen by heavy braided flexible leads attached to the blocks. In each case the framework holding the specimen is retained in position above a quench tank by supporting pins which are automatically withdrawn by solenoids at the completion of the heating period, allowing the specimens to fall freely under gravity into the quench tank.

The various heating and quenching operations to be carried out are controlled by a condenser-type process timer which consecutively makes and breaks two separate circuits, the time for which each circuit is made being controlled by two variable resistances. This technique for controlling heating cycles was first

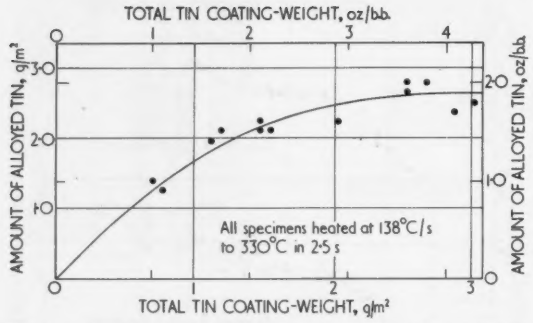

TOTAL TIN COATING-WEIGHT, oz/bb.
AMOUNT OF ALLOYED TIN, g/m²
AMOUNT OF ALLOYED TIN, oz/bb.
All specimens heated at 138°C/s
to 330°C in 2·5 s
TOTAL TIN COATING-WEIGHT, g/m²

Fig. 3—Formation of alloy in very thin tin coatings

used by Bailey and Watkins[10] for experiments on the flow of solders on metal surfaces.

CONCLUSIONS

On momentarily energizing the process timer a series of relays are operated in the first timed circuit which cause current to flow through a multi-tapped step-down transformer to the specimen. In the smaller unit a variable auto-transformer is interconnected to the primary of the step-down transformer so that the heating current may be varied at will. At the end of the heating period, the second timed circuit relays are made and energize the solenoids which release the specimen into the quench tank. With the smaller unit, by making suitable connections on the control panel, it is possible to reduce the current during this second period so that the specimen temperature remains constant for a known time before quenching.

Since with this type of process timer there is a slight drift about the pre-set time, the actual time for which the first (heating) circuit is made is measured by a relay-operated stop-watch, actuated simultaneously with the timer relays. The heating current passing through the specimen is measured by a current transformer placed in one of the heavy leads to the specimen.

The small flow-brightening unit together with its associated transformer is contained in a wooden box with hinged Perspex front for easy access, and maintained at 30° C by a thermostat. This ensures a constant electrical resistance in the transformer before heating of the latter is begun.

Before flow-brightening, the plated specimens were cut to size and treated cathodically in the hot alkaline cleaner for 1 min at 50 A/ft². This treatment was found to be necessary to ensure that there were no oxide films present which had formed during storage and would interfere with the brightening of the tin coating.

The smaller samples were mounted in the flow-brightening unit and allowed to stand in the constant temperature box for 20 min before heating.

Temperature Measurement

It was found by preliminary trials that to obtain accurate measurements of the peak temperatures of specimens heated at about 140° C/s, it was essential to employ a thermocouple of low heat capacity in intimate contact with the heated specimen and connected to a galvanometer of short period.

The technique finally perfected involved spot-welding 36 s.w.g. iron–constantan thermocouple wires in the required position on the specimen, one wire being welded on to each face.[11] The necessary welding current was supplied by the flow-brightening control panel to a specially designed jig with spring-loaded electrodes. The thermocouple leads were connected to a critically damped light-spot galvanometer having a period of 0·1 s for full-scale deflection. To obtain a permanent record of the maximum galvanometer reading, a strip of chlorobromide photographic paper was held firmly against the scale by a light-proof backing plate, so that the scale numbers were printed out in reverse and the maximum reading of the light-spot recorded. With the lower rates of heating a visual record of the galvanometer reading was easily obtained.

In most instances, the thermocouple junction was cut from the flow-brightened sample and calibrated in a small air-furnace against a sub-standard mercury-in-glass thermometer, and an average calibration curve was obtained for all the thermocouples used.

EXPERIMENTAL RESULTS

Alloy Coating-weight Measurements

The first group of experiments was concerned with the rate of formation of alloy and with the effect of total tin coating-weight under heating conditions similar to those existing in a commercial electrotinplate line.

A large number of samples were plated in the circulation cell to give four grades of materials having mean total tin coating-weights of 4·0, 8·1, 11·8, and

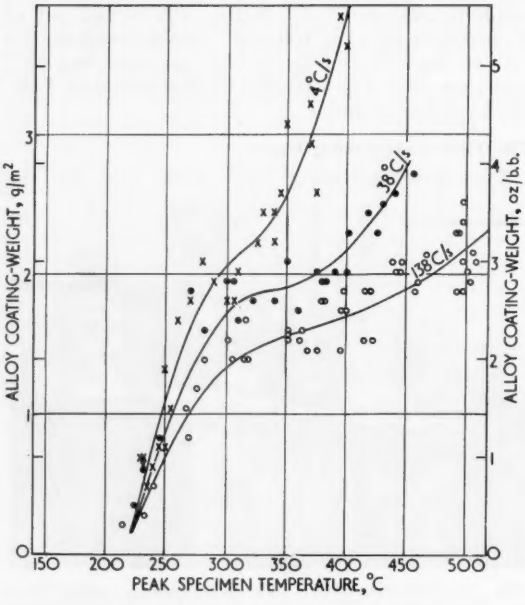


Fig. 4—Relationship between amount of alloy and peak specimen temperature for three rates of heating

15·7 oz/b.b. (2·8, 5·7, 8·3, and 11·0 g/m² respectively).* The specimens were heated at 138° C/s for pre-set times, the peak temperature attained by each sample being recorded. The heating times used covered the approximate temperature range 200–500° C, but a bright finish was only obtained within the range 260–380° C. Three alloy coating-weight determinations were made on each sample and in addition, nine readings of the reflectivity were taken as described below.

In Fig. 2 the alloy coating-weight is plotted against the peak specimen temperature for the four grades of materials used. The results shown in Fig. 2 indicate that there is a slight increase in the amount of alloy with total tin coating-weight, for any given flow-brightening temperature. This effect increases with temperature and is not significant at temperatures within the range normally used in a tinplate line.

Alloy formation in very thin tin coatings was also studied under conditions when the majority of the tin coating was converted to alloy, i.e. for total tin coating-weights up to about 4 oz/b.b.

Figure 3 shows the relationship between total tin and *alloyed tin* obtained from samples flow-brightened for a time, which would produce about 2·0 oz/b.b. of *alloy* in cases where excess tin was present. The heating rate was 138° C/s.

The second group of experiments was concerned with the effect of the rate of heating on the amount of alloy formed, and with the effect of holding for fixed times at a given temperature. All the samples used had a nominal coating weight of 8 oz/b.b.

The samples were heated under various conditions and in each case the total heating time, peak specimen temperature, and alloy coating-weight were recorded.

* The tin and tin–iron alloy coating-weight values given in the paper are expressed in the commercial units in common use in the U.K., i.e. ounces per basis box (oz/b.b.), and the following conversion factors may be applied:

1 oz/b.b. is equivalent to 0·0625 lb/b.b.
0·701 g/m²

In the various figures both the oz/b.b. and g/m² units are used for convenience.

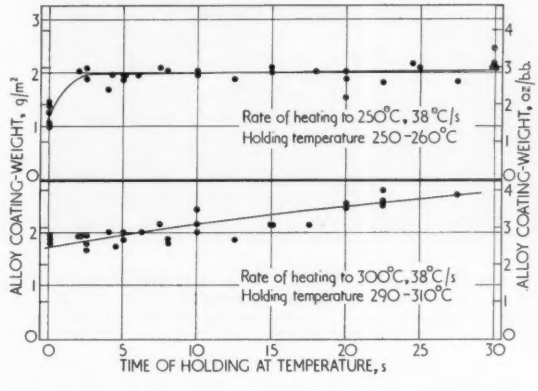

ALLOY COATING-WEIGHT, g/m²
ALLOY COATING-WEIGHT, oz/b.b.
Rate of heating to 250°C, 38 °C/s
Holding temperature 250–260°C
Rate of heating to 300°C, 38°C/s
Holding temperature 290–310°C
TIME OF HOLDING AT TEMPERATURE, s

Fig. 5—Relationship between amount of alloy and time of holding at constant temperature

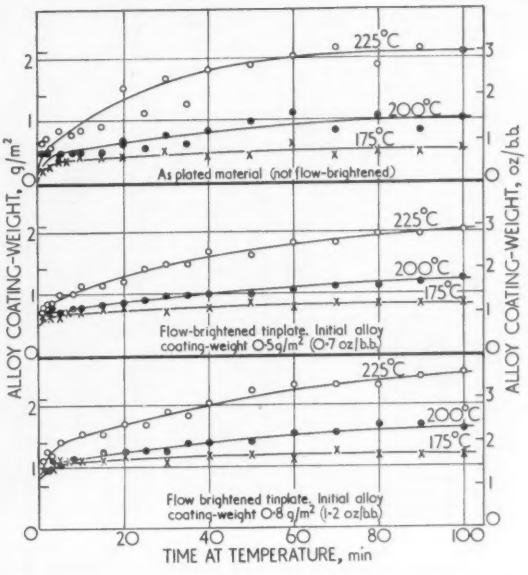

225°C
200°C
175°C
As plated material (not flow-brightened)
Flow-brightened tinplate. Initial alloy coating-weight 0·5 g/m² (0·7 oz/b.b.)
Flow brightened tinplate. Initial alloy coating-weight 0·8 g/m² (1·2 oz/b.b.)
ALLOY COATING-WEIGHT, g/m²
ALLOY COATING-WEIGHT, oz/b.b.
TIME AT TEMPERATURE, min

Fig. 6—Effect of heat-treatment below 232° C on amount of alloy

Curves obtained at heating rates of 138°, 38°, and 4° C/s are shown in Fig. 4, where alloy coating-weight is plotted against temperature. Other curves obtained by heating at intermediate rates have been omitted for clarity. At temperatures in the region 230–300° C the curves are only slightly divergent, but at higher temperatures there is a pronounced effect of heating rate, the inflexion obtained when heating at 138° C/s becoming progressively smaller.

In further experiments, samples were heated at the rate of 38° C/s, and then were held for periods up to 30 s within the ranges 250–260° or 295–310° C before being quenched. Figure 5 shows the rate of growth of the alloy layer produced by these treatments, and it is of interest that there is virtually no increase in alloy coating-weight after a period of 2·5 s at the lower temperature, whereas at the higher temperature the amount of alloy increases slowly but steadily during the entire holding period.

Alloy coating-weight determinations and metallographic examination of samples heated to about 200–230° C indicated the existence of traces of alloy, and experiments were therefore made to investigate the rate of formation below the melting point of tin. In addition to using as-plated material, it was decided to include specimens already having an alloy layer so as to simulate the stoving, at temperatures in the region of 200° C, of lacquered flow-brightened electrotinplate. This material was obtained from the centres of sheets of commercial electro-tinplate having known and uniform alloy coating-weights of about 0·7 and 1·2 oz/b.b.

The specimens were immersed in an oil-bath for periods up to about 2 h at temperatures of 175°, 200°, and 225° C, and Fig. 6 shows the resultant increases in alloy coating-weight with time.

Fig. 7—Not flow-brightened. Grain boundary etch on steel surface after pickling for 2 s in 2% H_2SO_4 at 85° C

Fig. 8—Heated to 200° C. Alloy coating-weight less than 0·3 oz/b.b. Beginning of alloying is not uniform

Fig. 9—Heated to 220° C. Alloy coating-weight 0·7 oz/b.b. Cementite in steel base still visible

Metallographic Examination

Cross-sections

Since the alloy layer in tinplate averages in thickness only about 3–12 μin., it was necessary to use taper-sectioning to obtain additional magnification to facilitate microscopic examination. Attempts were made to reveal the structure of the alloy by etching such micro-sections but with little success, and taper-sectioning was abandoned in favour of the simpler technique described below.

The Alloy–Tin Interface

The surface of the alloy layer was revealed by selective anodic dissolution of the free-tin layer in

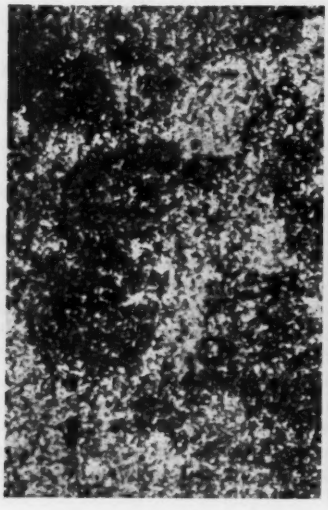


Fig. 10—Heated to 275° C. Alloy coating-weight 1·7 oz/b.b. Characteristic patchwork effect produced by varied orientation of underlying ferrite grains

Fig. 11—Heated to 400° C. Alloy coating-weight 2·7 oz/b.b. Patchwork effect much less than in Fig. 10

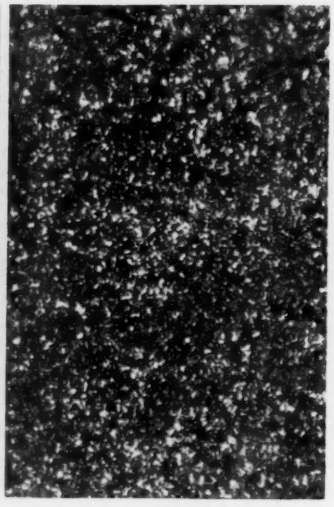


Fig. 12—Heated to 440° C. Alloy coating-weight 3·0 oz/b.b. Uniform appearance of alloy layer

Figs. 7–12 show the surface appearance of the alloy layer on flow-brightened specimens after removal of the free-tin layer × 1000

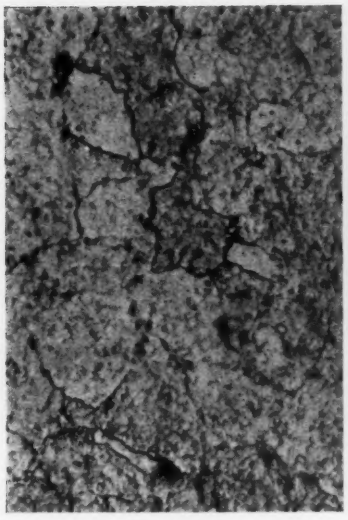


Fig. 13—Isolated patches of alloy at steel grain boundaries in regions where no fusion of tin occurred

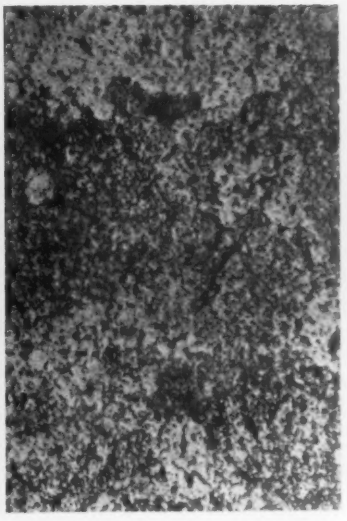


Fig. 14—Patches of alloy as in Fig. 13 becoming obscured by general alloying where tin coating has fused

Figs. 13 and 14 show at a magnification of × 2000 the surface of a specially prepared steel sample after plating, flow-brightening, and anodic removal of the tin coating

5% sodium hydroxide.[13] This method removes the free-tin layer and leaves the alloy unattacked.

Figures 7–14 show typical structures of the tinplate steel base and various amounts of alloy. The ferrite grains of the steel base produce a patchwork effect on the alloy layer when the latter has a coating weight of less than about 3 oz/b.b. This influence of the ferrite has been suggested by previous workers[6] to be due to different rates of alloy formation, depending on the orientation of the ferrite grains. The cementite particles in the steel base become invisible with amounts of alloy of 1·7 oz/b.b., or more. Specimens heated above 496° C, at which temperature $FeSn_2$ is converted to FeSn, showed little structural evidence of the transition having occurred. However, the surface of the alloy did not show its normal characteristic grey colour but appeared to have a brown tinge.

A comparison of the samples having similar amounts of alloy produced by heating under various conditions revealed that the microscopic appearance of the alloy surface was dependent mainly upon its thickness and

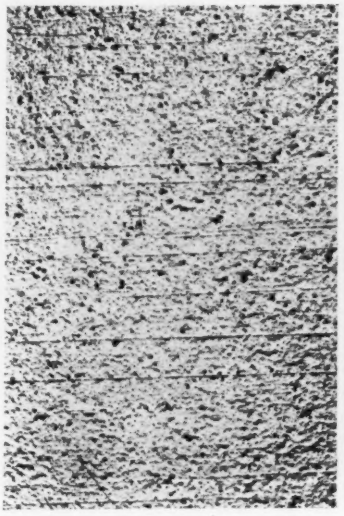


Fig. 15—Nominal 4 oz/b.b. specimen. No de-wetting of tin coating after heating to 400° C × 7

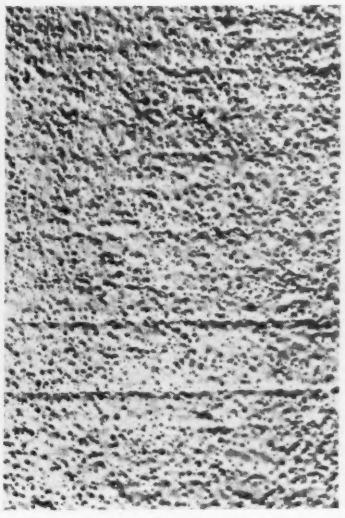


Fig. 16—Nominal 12 oz/b.b. specimen heated to 235° C. Slight de-wetting of tin coating × 7

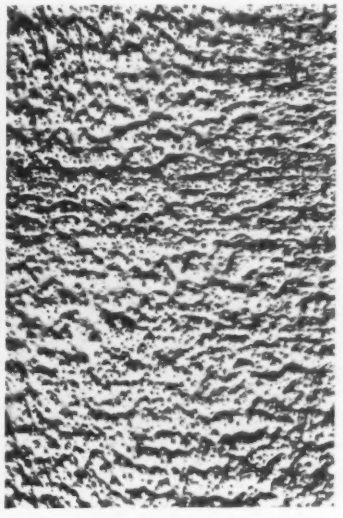


Fig. 17—Nominal 12 oz/b.b. specimen heated to 400° C. Severe de-wetting of tin coating × 7

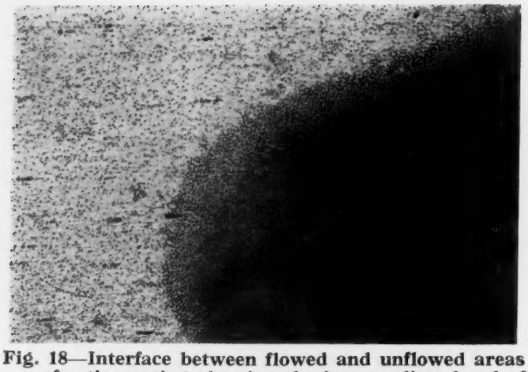


Fig. 18—Interface between flowed and unflowed areas of a tin coating showing the intermediate band of semi-bright tin × 3

not on the heating conditions under which it was formed.

Examination of samples heated into the range 200–232° C showed isolated patches of alloy situated generally within the ferrite grains (Fig. 8), and it was difficult to establish whether alloying commenced within the grains or at the ferrite grain boundaries because the tinplate steel base retained a deep boundary etch from the manufacturing process. To examine the point further, some samples of steel were given 2–3% reduction between polished rolls and were then metallographically polished using diamond paste. A short etch was given in 2% nitric acid in alcohol and the specimens were subsequently plated in the circulation cell and flow-brightened so that only part of the tin coating melted. In the regions where no fusion had occurred, small areas of a phase which had the appearance of $FeSn_2$ were found at the ferrite grain boundaries, principally at the intersection of several boundaries (Fig. 13). In regions where the tin coating had melted, general alloying had occurred in all the ferrite grains and the original patches in the grain boundaries were obscured (Fig. 14).

The Tin Coating Surface

Examination of the surface of flow-brightened samples of tinplate showed that in the nominal 4 and 8 oz/b.b. materials there was little or no ' de-wetting '* of the tin coating even after heating to 400° C (Fig. 15). In the nominal 12 oz/b.b. samples the extent of de-wetting increased with flow-brightening temperature, as shown in Figs. 16 and 17, while with the 16 oz/b.b. specimens a similar effect was observed but was more pronounced.

* ' De-wetting ' is the condition where the tin coating does not remain uniform during fusion but coalesces into individual globules leaving the intervening areas devoid of any appreciable amount of free tin.

Fig. 19—As-plated tin coating × 200

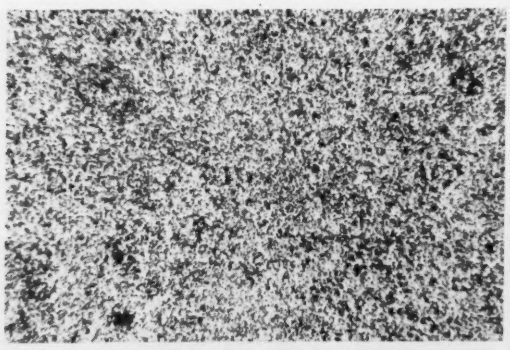
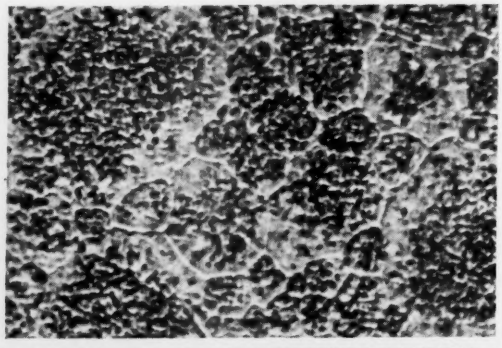


Fig. 20—Beginning of melting of tin coating forming ' grains ' × 1000



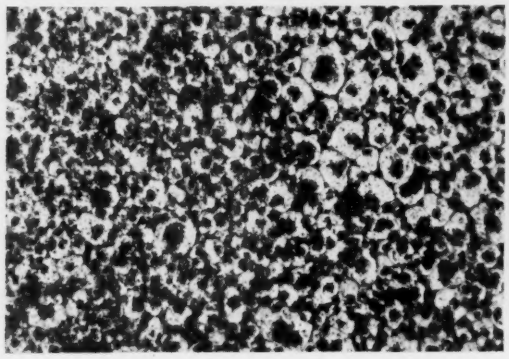


Fig. 21—More extensive melting of tin coating than in Fig. 20 leaving isolated areas unfused in centres of tin ' grains '. Extent of melting increases from right to left × 200

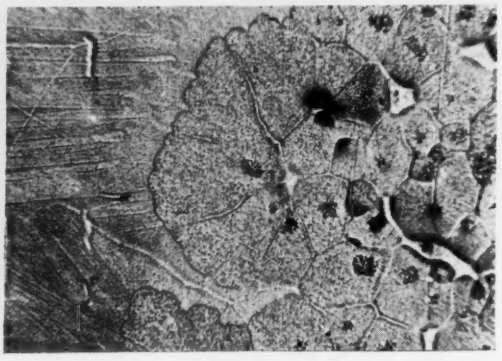


Fig. 22—Almost complete fusion of tin coating with (at left) coalescence of tin ' grains ' to form a uniform melted coating × 200

Figs. 19–22 show the differences in appearance of the tin coating surface between the fused and unfused regions of an incompletely flowed sample

Microscopic examination of the flowed/unflowed interface in partially melted specimens revealed a narrow intermediate band of semi-bright material (Fig. 18). At a higher magnification, the transition from as-plated to fully-melted tin was shown clearly, and Figs. 19–22 were taken successively across this region. Melting of the tin coating occurs initially on lines forming ' grains.' These grain boundaries were found in most cases not to correspond to the underlying ferrite grain boundaries. These regions of melted tin gradually spread and absorb the enclosed areas of unmelted tin and the tin ' grains ' finally coalesce to form a uniform melted layer. This forms, on quenching, the relatively large feather-shaped crystals which are a characteristic of the conditions obtaining in the present apparatus.

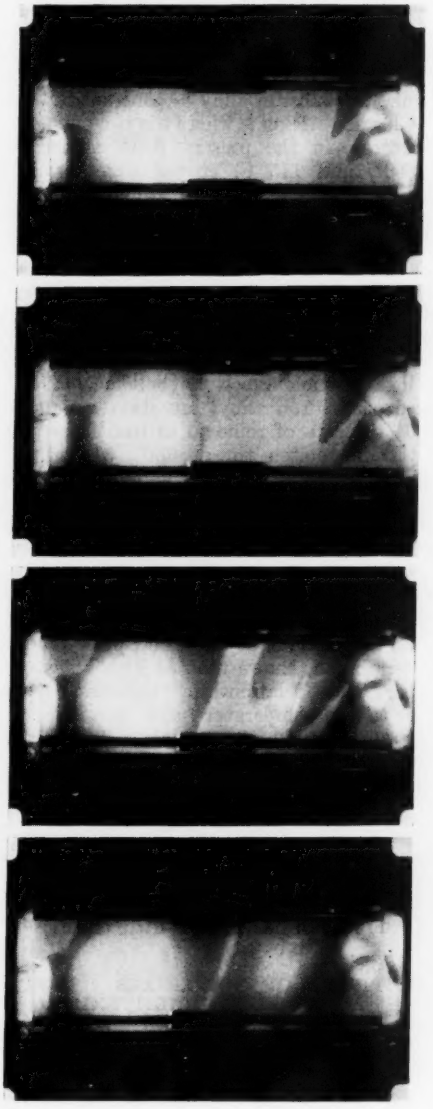


Fig. 23—Four frames from film taken at 1000 frames per second of melting of tin coating. The melted tin appears dark and the unflowed regions are light in colour. The circular white area on the left is a reflection of the spot light

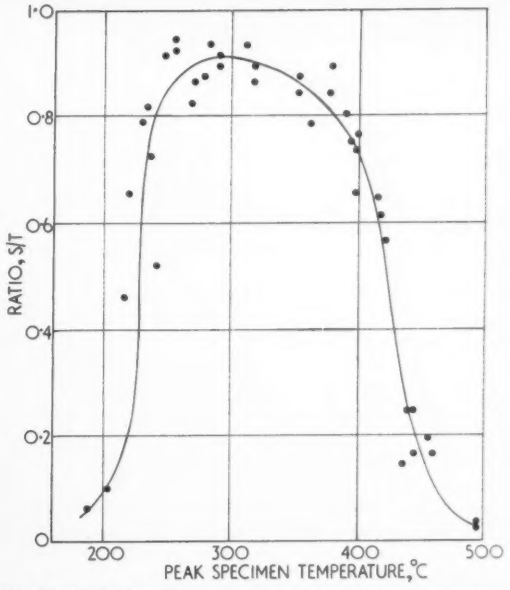


Fig. 24—Relationship between peak specimen temperature and reflectivity for nominal 8 oz/b.b. specimens

The actual melting of the tin coating on large specimens was observed by the technique of high-speed cinematography. Films were taken at speeds of 500 and 1000 frames per second, at two camera distances. The movements of the liquid/solid tin interface were observed, and it was found that there was a tendency for the melting to occur in ' bursts,' i.e. the interface appeared to halt periodically during its rapid movement.

Generally, melting occurred from the end of the samples and the time for the tin coating to melt was about 0·1 s. Four successive but not adjacent frames taken from the 1000 frames per second film are shown in Fig. 23 to illustrate the mode of melting of the tin coating. In one instance melting occurred simultaneously from about six points throughout the sample so that the complete fusion cycle occupied, at the most, 0·05 s.

A feature shown by each film was a marked increase in reflectivity of the tin coating in the period before melting occurred. There is no obvious explanation of this phenomenon but it may be connected with the formation of traces of alloy below the melting point of tin.

The tin coating of many samples was etched in hydrochloric acid and it was found that there was a marked increase in grain size with flow-brightening temperature, although the grain sizes and shapes encountered were not of the same order as those encountered in commercial tinplate.

Reflectivity

A modified Guild instrument was used to measure the reflectivity of many of the flow-brightened speci-

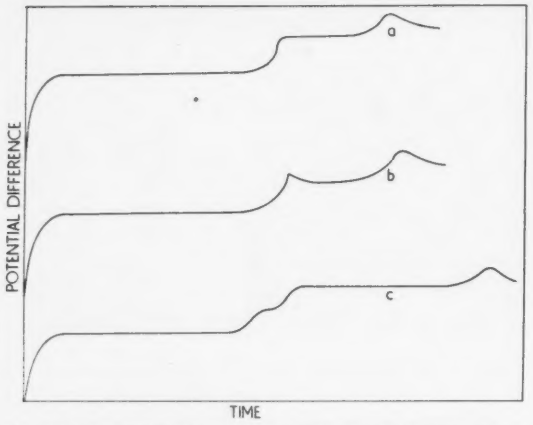


Fig. 25—Potential/time curves obtained during alloy coating-weight determinations

mens. This instrument measures the ratio (S/T) of the specularly reflected light (S) to the total light reflected and diffused (T), and hence indicates the surface smoothness of the material.

Curves were plotted of S/T against flow-brightening temperature for most of the large specimens and a typical curve, the one obtained with the nominal 8 oz/b.b. material, is shown in Fig. 24. The reflectivity reaches a peak value and then falls rapidly, owing to oxidation of the tin surface. The peak value was slightly lower for the nominal 4 oz/b.b. samples than for the three heavier coating-weights. The S/T ratio of the samples not flow-brightened was practically the same for the 8, 12, and 16 oz/b.b. materials, but was considerably higher for the 4 oz/b.b. tinplate, presumably owing to the influence of the steel base, which has a high S/T ratio, (0·62). These points are shown in Table II.

Potential Measurements

During the course of the routine alloy coating-weight determinations, the shapes of the potential/time curves plotted by this technique showed features of some interest.

As described in a previous paper,[8] the normal type of curve was that shown in Fig. 25*a* where the amount of alloy was about 2·0 oz/b.b. Increasing temperature of flow-brightening caused an increase in the alloy coating-weight, and at values in the range 3·0–3·5 oz/b.b. other types of curves were obtained (Fig. 25*b* and *c*). It appeared that these curves were only obtained when the temperature reached by the specimen was about 500–550° C, which is above the transition temperature of the alloy. Examination of the steady potential values showed no definite evidence of the presence of any compound other than $FeSn_2$, although the extra inflexion in the curve (Fig. 25*c*) may indicate the presence of another phase.

The cause of the peak at the beginning of the dissolution of the alloy (Fig. 25*b*), which is not obtained with low alloy coating-weights, is inexplicable; although Kunze and Willey and other workers obtain this type of curve as a normal practice during routine coating-weight determinations and it may be a characteristic of the experimental conditions.

DISCUSSION

It is interesting that, in the present tests, materials aged at temperatures below the melting point of tin for short periods showed a measurable increase in the amount of alloy present. For example, in the as-plated samples, ageing for 1 min at 225°, 200°, and 175° C produced, respectively, alloy coating-weights of 0·9, 0·7, and 0·3 oz/b.b. In addition, specimens heated in the large unit to temperatures of about 225° C showed alloy coating weights of about 0·3–0·7 oz/b.b. On the contrary, Davies and Hoare[6] found no traces of alloy after heating as-plated samples for periods of 20 s at 229° C, but it is likely that the amounts of alloy likely to be present escaped detection by the methods then used.

It is reasonable to assume that the rate of formation of $FeSn_2$ obeys the law relating to a diffusion reaction, i.e. thickness of alloy = constant $\times\sqrt{}$time or $\log d = \log K + \frac{1}{2}\log t$, where t is time at any given temperature. In the present tests carried out at 225°, 200°, and 175° C, curves were obtained by plotting $\log d$ against $\log t$ which had positive slopes increasing slowly with time. For practical purposes the relationships are virtually linear for periods up to about 20 min but over the whole period of time investigated (100 min) the diffusion equation is apparently not obeyed. On the contrary, the results of similar tests carried out by Kunze and Willey[7] gave straight-line relationships when plotted in the above logarithmic manner. Seybolt[2] also investigated the rate of tin–iron alloy formation by an indirect method; the rate of removal of iron from a sample immersed in molten tin at 330°, 315°, 290°, and 260° C was measured by a magnetic method. Seybolt concluded that the rate of attack on the iron obeyed the parabolic diffusion law, but at 260° C there is a deviation, for the shorter times of immersion, resulting in a curve similar to those obtained with the present tests.

The effect of rate of heating on the relationship between the amount of alloy and peak temperature of the specimen, shows that at high rates of about 140° C/s, the relationship becomes practically parallel to the temperature axis over the range of 300–400° C (Fig. 2). This means that within this range the amount of alloy obtained is virtually constant, whereas decreasing the rate of heating to about 4° C/s tends to eliminate this flat portion of the relationship. Owing to the limitations of the apparatus the effect of rates of heating in excess of 140° C/s was not investigated but might lead to some interesting results. The overall effect is that below 300° C the rate of heating has little effect.

Table II

REFLECTIVITY OF MATERIALS BEFORE AND AFTER FLOW-BRIGHTENING

Nominal Tin Coating Weight, oz/b.b.	S/T Ratio As-plated	Peak S/T Ratio after Flow-brightening
4	0·38	0·85
8	0·08	0·91
12	0·11	0·93
16	0·11	0·94

Microscopic examination of many samples, after removal of the free-tin layer, did not readily reveal in which regions alloying first took place, but by the use of steel samples, which had been given a metallographic polish and etch before plating and flow-brightening, it was observed that isolated areas of what was thought to be $FeSn_2$ were situated at the junctions of the ferrite grain boundaries (Fig. 13). This phenomenon was confined to those regions close to the solid/liquid interface where the tin coating had not quite melted. However, one might expect that formation of the alloy would be most rapid in the centres of grains where the total energy is highest, and indeed, in the regions where the tin coating had just melted the initial traces of alloy were invariably distributed as shown in Fig. 8, i.e. in non-uniform patches. The patchwork effect of the alloy was at first considered to be accentuated by the way in which the tin coating melted, that is, the joining-up of melted regions of the tin to form 'grains,' as described earlier (Fig. 20). However, there appeared to be no distinct pattern formed by the alloy similar to that of the melted regions of tin. It was also found that only in a few instances did these tin 'grains' correspond with the grains of ferrite in the steel base and there appears to be no obvious explanation of this peculiar manner of melting of the tin coating. It is considered possible that this phenomenon may be connected with the co-deposition of the addition agent from the electrolyte during the plating operation.

Surface examination of samples at low magnifications revealed that prolonged heating of nominal 4 and 8 oz/b.b. coatings, either with constant or increasing temperature, produced little or no de-wetting. It is well known in practice that 12 and 16 oz/b.b. coatings are much more liable to de-wet during flow-brightening, and in the former material it was found that heating to 235° C caused little or no de-wetting, while in the latter material de-wetting apparently occurred as soon as the coating melted and was pronounced when heating was prolonged. Other work has shown that the extent of this phenomenon is dependent, to a certain extent, on the surface preparation of the steel before plating, and for thicker tin coatings, a severe anodic etch has been found to considerably reduce the tendency to de-wet.

Concerning the general surface appearance, it was noticeable that the 12 oz/b.b. material consistently gave the most brilliant and pleasing appearance to the naked eye when compared with the other three grades. However, the measurements made with the reflectometer showed no evidence of this fact, and the human eye is certainly able to detect certain intrinsic properties of a surface other than surface smoothness.

In samples heated to above 496° C where the reaction $FeSn_2 \rightleftharpoons FeSn$ occurs, the potential time curves obtained were of the type shown in Fig. 25*c*. Although there was little metallographic evidence of the transition having taken place, these curves suggested the presence of a phase situated between the free-tin and $FeSn_2$ layers. It is suggested that this phase is FeSn and a possible explanation of its presence at the tin/$FeSn_2$ interface and not on the iron-rich interface may be as follows: during heating to above 496° C, $FeSn_2$ is formed and changes to FeSn but during cooling the reverse reaction is prevented only in those parts of the alloy layer where the quench is most drastic, that is, at the outer surface. However, this explanation is not supported by the previous work of Jones and Hoare[5] who found that the phase FeSn was readily retained even during furnace cooling from above the transition temperature.

CONCLUSIONS

The relationships between alloy coating-weight, time, and temperature, during the flow-brightening of electro-tinplate, has been studied in conjunction with extensive metallographic examination of the resultant alloy layer. The results have been compared with those of other workers and some points of similarity were obtained as well as some contradictions. The effect of flow-brightening conditions on the surface of the tin coating has been studied, both microscopically and in relation to the brightness and general appearance of the tinplate.

It may be said that, generally, the alloying of tin and iron during flow-brightening proceeds very rapidly, and control of the amount of alloy formed can only be obtained by accurate temperature and time control during the operation. In addition, it should be borne in mind that appreciable alloying occurs below the melting point of tin, both in the heating-up period before the fusion of the coating, and also during relatively long-term heat-treatments, such as the stoving of lacquered tinplates.

Acknowledgments

The author wishes to thank Dr. W. E. Hoare and his colleague, Mr. R. Mills, for many helpful discussions, and Miss N. Young who assisted with much of the laboratory work. This work, which was carried out in the laboratories of the Tin Research Institute, forms part of the tinplate research programme of the International Tin Research Council, to whom the author is indebted for permission to publish.

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Formation and Tempering of Martensite in 18/8 Steels

By Paul G. Bastien, D.Sc., and Jacques M. B. Dedieu, Dr.-Ing.

SYNOPSIS

The possibility of bringing about a considerable amount of $\gamma \rightarrow \alpha$ (martensitic) transformation in low-carbon 18/8 Cr–Ni steels has been investigated experimentally.

Plain cooling to very low temperatures (down to the b.p. of liquid hydrogen) produces transformation of only a small portion of the austenite. On the other hand, plastic straining at temperatures slightly below or above the ordinary does produce a considerable amount of the α-phase. Strain-hardening, so characteristic of 18/8 steels, is in no way diminished by the possibility of transformation during working, but in some cases is actually increased.

Dilatometric and X-ray tests have demonstrated that the α-phase developed in a steel by straining plastically disappears if the steel is reheated at a temperature exceeding 550° C. The rate of disappearance of the α phase is rapid between 550° and about 650° C, but the final stages of the reaction between 650° and 750° C are relatively slow.

Reheating at temperatures up to 500° C has no effect on steels previously transformed almost completely to α-phase by plastic straining, but reheating at temperatures above this causes a progressive loss of hardness. The hardness of partially transformed samples is increased by reheating at temperatures up to about 500° C, the amount of the increase depending upon the chemical composition of the steel and the conditions of the straining as well as the reheating temperature. 1179

MARTENSITE OBTAINED BY SIMPLE COOLING

THE PRESENT AUTHORS have reported[1] the results obtainable by quenching 18/8 Cr–Ni steels in various media after heating to 1050–1100° C. A part of the austenite transforms into the α-phase; the kinetics of this process are martensitic in character. In all cases, it is observed that with plain cooling, without any cold working, the transformation starts only at very low temperatures and only becomes notable at temperatures near absolute zero, a circumstance which considerably reduces the practical value of such treatment.

In addition, under such conditions, *the yield of the transformation is quite small.* Thus the specific saturation magnetization of samples quenched to different temperatures has been determined and the ratio of these values to those for the fully ferromagnetic condition has been derived (*see* p. 256 for the absolute specific saturation values). These ratios measure the proportion of the α-phase obtained in the samples by the martensitic transformation, one of the characteristics of which is that it involves no change of composition. Figure 1 is a plot of the fraction transformed at different temperatures in a steel of composition: C, 0·044%; Cr, 18·8%; Ni, 7·99%, obtained by extrapolating to infinite field strength the magnetization curves given by a magnetometer operating at field strengths of up to 20,000 oersteds.* It shows that in this steel, which is particularly prone to transformation, one cannot hope to obtain more than 15% of the α-phase, even at absolute zero.

To appreciate the full significance of this, it must be borne in mind that the onset of transformation is essentially a function of the nickel content,[2] and that it rises by 50° C for every 1% decrease in nickel content. However, for present-day 18/8 steels, the amount of the α-phase thus obtained is too small to

* Pauthenet magnetometer kindly provided by Professor Neel, of Grenoble.

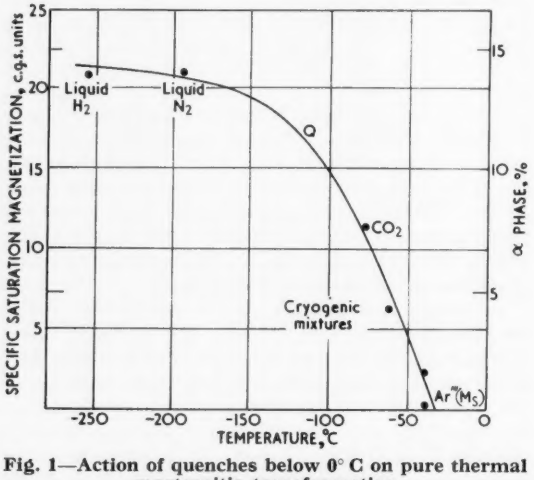

Fig. 1—Action of quenches below 0° C on pure thermal martensitic transformation

Manuscript received 18th August, 1954.

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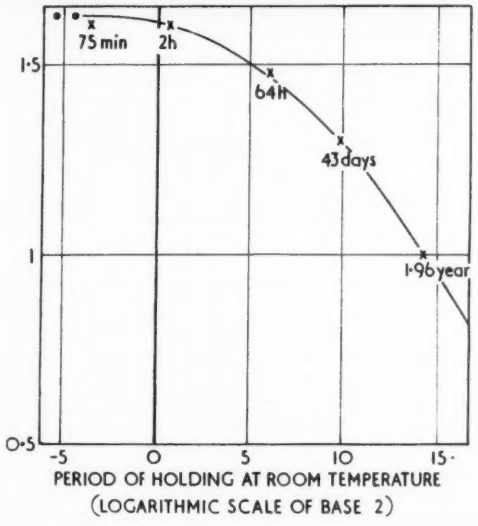

Fig. 2—Effect of stabilization on quench in liquid nitrogen

be of great interest: the properties, at ordinary temperatures, of 18/8 steels quenched in liquid nitrogen or hydrogen are still very close to those of the same steels superquenched in the austenitic state.

Another limitation of the method is connected with a form of stabilization. Under practical conditions, samples which may have to be cooled eventually may be left at ordinary temperatures for some time. Under such conditions, it is found that the yield in the transformation decreases with the time of holding at ordinary temperature. Figure 2 relates to a steel containing 0·04% C, 18·8% Cr, and 9·04% Ni; the ordinates are divided in units of magnetic attraction for a magnetic balance of the authors' design[3] and the abscissa is a logarithmic scale of the period of stabilization.

MARTENSITE OBTAINED BY PLASTIC STRAINING

On the other hand, depending on the chemical composition, plastic straining at ordinary tempera-

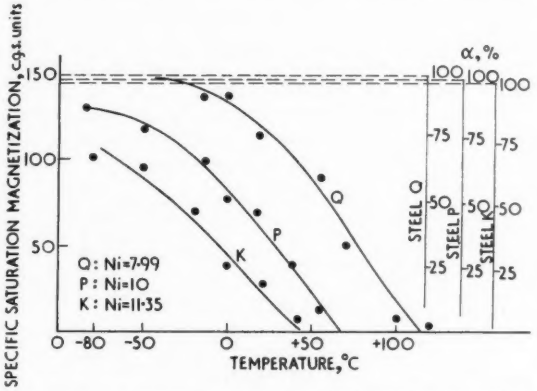

Fig. 3—$\gamma \rightarrow \alpha$ transformation by 50% height reduction in up-ending test

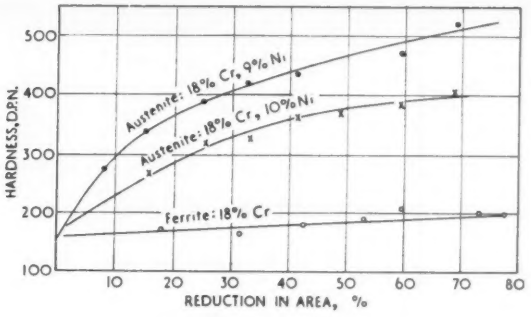

Fig. 4—Variation of Vickers hardness with plastic straining

ture (or at temperatures of up to 200° C on either side) produces a martensitic transformation in practically all the 18/8 steels studied.

The authors have described elsewhere the result of magnetic measurements on a bar drawn through a wire-drawing die: 77·4% reduction of cross-sectional area produces 83·5% transformation of austenite to the α-phase in a steel of composition near to that just quoted.[4]

The effect of temperature emerges very clearly from a series of experiments made in a thermostat,[3] in which small cylindrical samples 8 mm high × 8 mm dia. were subjected to an upending test involving 50% reduction in height. The straining induced by this treatment is revealed by the appearance of magnetism, which is measured in terms of the specific saturation. Figure 3 shows curves for three steels chosen for their difference in nickel content:

Steel	C, %	N, %	Ni, %	Cr, %	Mn, %
Q	0·044	0·031	7·99	18·8	0·34
P	0·035	0·028	10	18·3	0·41
K	0·031	0·031	11·36	18·6	0·40

The left-hand scale indicates the magnetic saturation values obtained by extrapolating to infinite field strength the magnetization results obtained using magnetic fields of up to 20,000 oersteds, and the right-hand scale indicates the corresponding content of α-phase. Comparison of the family of curves with the single curve in Fig. 1 (no plastic straining) shows up very sharply the difference in yield of the two methods. Steel *Q* has the same composition as the steel on which Fig. 1 is based, but upsetting it at temperatures below − 50° C brings about almost complete transformation. At 0° C more than 75% of the austenite transforms.

The effect of nickel is to displace the curves towards lower temperatures.

The points at which the curves meet the abscissa correspond to the temperature of initiation of martensitic transformation under the particular experimental conditions adopted. It will be noted that all these temperatures are higher than room temperature and higher than that induced by simple quenching. The latter is, of course, the conventional M_s temperature, usually referred to in France as the A_r'''.

Does a martensitic transformation by plastic straining affect the mechanical properties of 18/8

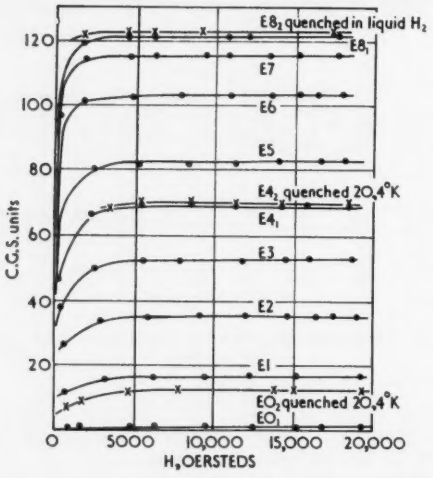

Fig. 5—Stainless steel (18/8) cold-drawn to different percentage reductions

steels, particularly their hardness ? It is well known that cold work increases the hardness of non-transformed steels. Figure 4 presents a comparison of a 9% Ni steel, almost completely transformable by plastic straining (by drawing a solid bar through a die), with a 10% Ni steel which has not suffered transformation and a ferritic steel containing no nickel. The compositions are:

Steel	C, %	Cr, %	Ni, %
18/9	0·040	17·8	9
18/10	0·035	18·3	10
18/0	0·05	18·02	0

The diagram shows that strain-hardening occurs just as much in the nickel steel which transforms as in that which does not. More generally, the processes of austenite hardening and martensitic transformation are both affected by the composition, and it is difficult to separate the two effects. However, it can be said that there is at least as much hardening in cases where there is transformation, with high degrees of deformation, as with steels which remain entirely austenitic. The effect is actually greater in the diagram shown in Fig. 4.

Stabilization of austenite by holding at room temperature does not seem to occur in steels subjected to plastic straining, in contrast with those subjected to plain quenching: no significant difference was found between the saturations measured on samples subjected to upending tests after holding room temperature for periods of from 1 h to 1 year.

This section concludes with some details about the magnetic quantities on which the investigation was based. The difficulty encountered in making exact quantitative measurements of the percentage of the α phase by the magnetic method should be emphasized, if very high magnetizing fields are not used. This emerges particularly from the curves for specific magnetization, $\sigma = f(H)$, obtained on ellipsoidal specimens supposed, for purposes of measurement, to be uniformly magnetic, and making allowance for the demagnetizing field: Fig. 5 gives an example for a

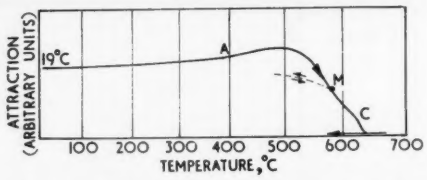

Fig. 6—Chevenard thermomagnetic test on transformed cold-drawn austenite

steel denoted by E, containing 0·04% C, 17·8% Cr, 9% Ni in which $E_0, E_1 \ldots E_8$ represent the superquenched states, which have suffered 0, 10%, 20% ... 80% reduction in cross-sectional area. It will be seen that when the proportion of α-phase present is small, the curves become amenable to extrapolation only at field strengths in excess of 5000 oersteds. This is satisfactorily explained by the presence of high internal demagnetizing fields due to the dispersion of the ferromagnetic phase in a relatively large amount of paramagnetic phase.

Finally, the theoretical values, determined by approximate calculation, of the absolute specific saturation for Fe–Cr–Ni alloys[3] corresponding to the range of compositions studied, supposing them to be entirely ferromagnetic (100% α-phase) are as follows:

18% Cr, 0% Ni: $\sigma_s = 167$ c.g.s. electromagnetic units
18% Cr, 4% Ni: $\sigma_s = 159$,, ,, ,,
18% Cr, 8% Ni: $\sigma_s = 151$,, ,, ,,
18% Cr, 12% Ni: $\sigma_s = 143$,, ,, ,,

The values of the absolute specific saturation of the ferromagnetic phase have been calculated (*a*) by determining the number of magnetons of the Fe–Cr group in the alloy; (*b*) by assuming that it has an electron concentration that lies between that of iron and that of nickel; and (*c*) by applying to the alloy the law of binary Fe–Ni mixtures based on a diagram due to Shockley—in fact, the Fe–Cr group has a very high iron content and probably behaves very like pure iron.

The values of the spontaneous magnetization at room temperature differ little from those for the absolute saturation. No correction has been made for temperature, since the law of variation of the saturation as a function of the ratio T/θ_c (T is the absolute temperature and θ_c the Curie point), indicates that only a slight error is introduced when $T/\theta_c < 0{\cdot}4$. For all the alloys used the Curie point is certainly above 600° C, as can be verified by means of the Chevenard thermomagnetometer, which shows that the α-phase can be magnetically detected right up to the start of the $\alpha \rightarrow \gamma$ transformation.

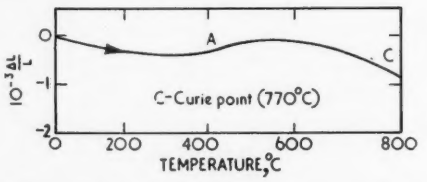

Fig. 7—Differential expansion in Chevenard apparatus for axial specimen of cold-drawn Armco iron

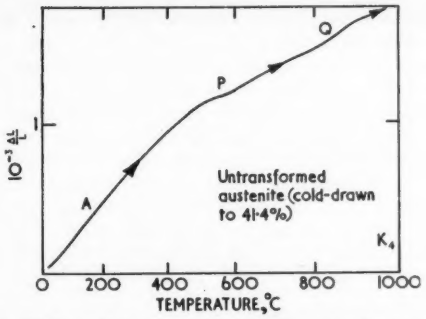

Fig. 8—Differential expansion in Chevenard apparatus for an untransformed austenite

PROCESSES INVOLVED IN TEMPERING 18/8 STEELS TRANSFORMED BY COLD WORK

Thermomagnetic Analysis

Thermomagnetometry by Chevenard's method[5] enables the variation in magnetization of wire samples with temperature to be followed. The curve obtained for a steel containing 0·04% C, 17·8% Cr, 9% Ni, cold-drawn at ordinary temperature to 77% reduction in area, using a test piece excised axially from the bar, is shown in Fig. 6. A small modification is succeeded by an increase of magnetization (A) which is then masked by a transformation in which the ferromagnetic phase progressively disappears irreversibly (M), while the magnetization falls rapidly to zero near the Curie point (C), which is recognized by the reversibility which it implies and which has been confirmed in the present investigation; the study of the transformation is abruptly halted by this appearance of the Curie point. However, if the specimens are heated a little above C, a partial magnetization reappears on cooling whose intensity is the lower the higher the temperature (above C).

The explanation of the change at A emerges from the present investigation: it arises from an increase of permeability through relief of stresses in the specimen; M probably indicates the onset of an $\alpha \rightarrow \gamma$ transformation starting at about 550° C.

Dilatometric Measurements

Chevenard's method,[6] which consists in recording the difference between the length of the test piece and a standard having uniform and reversible expansion as a function of temperature, was used. With a Pyros[6] standard, the differential expansion of an austenitic steel is positive, and that of a ferritic one negative. To obtain a detailed interpretation of the anomalies to be found in cold-worked and transformed steels, it is necessary to know the anomalies in a cold-worked sample entirely of α and one entirely of γ. Figures 7 and 8 relate to a ferritic steel with 0·05% C, 18·02% Cr, 0·17% Ni, 0·32% Mn, and 0·46% Si, cold-drawn as rod to 77% reduction in area, and to an austenitic steel, containing 0·033% C, 18·6% Cr, 11·3% Ni, cold-drawn to 41·4% reduction in area, untransformed and not appreciably ferromagnetic. In every case, the samples were taken along the axis of the rod. It will be observed that the slope of these curves characterises the α or γ state of the material, just as if the samples were in the annealed states. The anomalies arise from stress-relief (A) and Curie point (C) for the ferritic, and from stress-relief and weak carbide precipitation (P, Q) for the untransformed cold-drawn austenitic, steels.

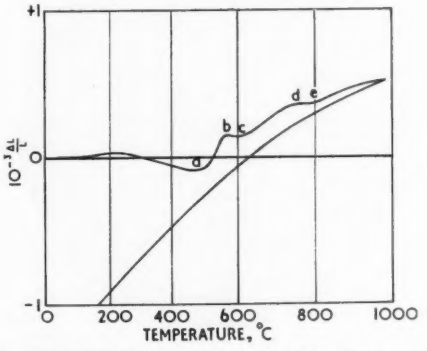

Fig. 9—Differential expansion of cold-drawn steel at room temperature to 63% reduction in area

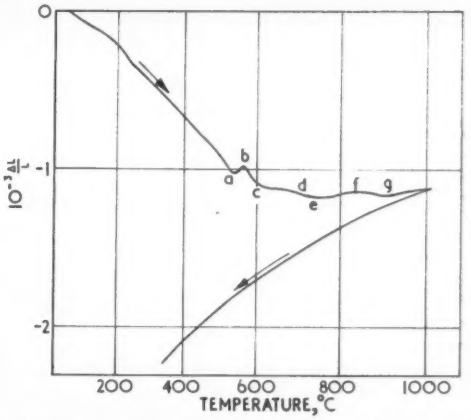

Fig. 10—Differential expansion of an 18/8 steel cold-drawn at room temp. to 77% reduction in area

Figure 9 is a differential dilatation curve for a steel containing 0·044% C, 18·8% Cr, 7·99% Ni, and 0·34% Mn, that had been previously transformed almost completely to α phase by cold drawing at room temperature (77% reduction in area). Figure 10 is the corresponding curve for another steel containing 0·04% C, 17·8% Cr, 9·04% Ni, also converted almost completely to α-phase by a similar cold-drawing treatment. A number of discontinuities, apparent in these curves, require comment. The first of these is located in the temperature range 200–300° C and is attributed to stress relief. Next comes the discontinuity marked *ab* on Figs. 9 and 10. Other experiments, details of which have been omitted in the interests of brevity, have shown that the reaction causing this discontinuity is rapid, limited to a very narrow temperature range and independent of the rate of heating, at least over the range 24–300° C/h. The peak effect appears to occur at about 580° C and is greatest for samples previously subjected to a reduction in area of 40–50%. Manifestations of the phenomenon responsible for the discontinuity *ab*

appear to be counteracted and limited in extent by the contraction ascribed to the $\alpha \rightarrow \gamma$ transformation (marked *bc* in Figs. 9 and 10).

Other discontinuities marked *c*, *d*, *e*, *f*, *g*, and *h* are considered to correspond to the gradual completion of the $\alpha \rightarrow \gamma$ transformation. The effects of carbide precipitation on the dilatation curves are not clear.

By its uniqueness and reproducibility, the anomaly *ab* appears to be most worthy of attention. Attempts to explain it in terms of a combination of processes known to occur in steels proved to be in vain: although there is no doubt whatever that it is followed by the development of γ phase and that it involves an expansion.

(i) Before *ab*, the expansion curve is reversible, return taking place along the slope of the cooling curve in the immediate neighbourhood of the point considered.

(ii) The expansion *ab* occurs in a zone which decreases in breadth as the initial plastic straining of the sample increases. The fact that its intensity passes through a maximum for 40–50% reduction in area suggests that the process *bc* (of the $\alpha \rightarrow \gamma$ type) increases in importance as the reduction in area increases, and that it limits both the amplitude and the temperature of the *ab* process.

(iii) Micrographic examination shows that, after quenching in water from temperatures in the range *ab*, the structure is more readily resolved than in cold-drawn samples and contains numerous small spheres resembling carbides. The number of these spheres visible is surprising considering the small amount of carbon in the steel.

The dilatometric studies reported very briefly above add the following information to that provided by the thermomagnetic studies: there is superimposed on the $\alpha \rightarrow \gamma$ process, within a narrow temperature range, an inexplicable process which causes an expansion. In addition, it has been observed that the $\alpha \rightarrow \gamma$ process (*bc*) leads to a considerable and rapid reduction of magnetism, which, however, does not entirely vanish until 680–700° C is attained, depending on the holding time.

Similar examples are known of the stepwise disappearance of the α phase in low-carbon steels, containing 18% Cr and 4–6% Ni. In this case, they correspond to the successive transformation of a martensitic phase, and then of a ferritic phase. The martensite transforms rapidly to austenite, but ferrite transforms only slowly because of the slow diffusion, notably of chromium atoms concentrated in it. It is probable that in the case with which we are concerned, the process is similar to the stabilization of retained α phase, caused by the diffusion of chromium, at high temperatures (650° C and above).

X-Ray Diffraction Study

Using unfiltered radiation from an iron anti-cathode produced from the cold-worked specimens a large number of lines on the Debye-Scherrer spectrum. After holding for 2 h at various tempering temperatures it could be observed that the less intense lines characteristic of the α phase disappeared. By observing closely the aspect and the intensity of darkening of the K_α and K_β lines and their disappearance, it became possible to place the $\alpha \rightarrow \gamma$ reaction with certainty between 520° and 720° C. Nevertheless, it is not possible by this method to fix very exactly either the start or the end of this transformation.

It was of interest to become better acquainted with the *ab* anomaly revealed by dilatometry. Without doubt, because it is localized in a very narrow temperature zone, it has escaped detection in most X-ray investigations made on samples heated to various temperatures, and yielding information only about isolated points. In most cases all that are observed are the lines corresponding to diffraction by the α or γ lattices. However, in several repetitions of the present tests on specimens heated to 580° C and thereabouts, it was noted that two cases of new lines corresponding to the following lattice distances, evaluated in kX units:

$d = 3 \cdot 05$, moderately weak	$d = 1 \cdot 94$, moderate
$d = 2 \cdot 64$, very weak	$d = 1 \cdot 88$, strong
$d = 2 \cdot 35$, strong	$d = 1 \cdot 59$, moderate
$d = 2 \cdot 24$, strong	$d = 1 \cdot 23$, moderately weak
$d = 1 \cdot 98$, very strong	$d = 0 \cdot 996$,,

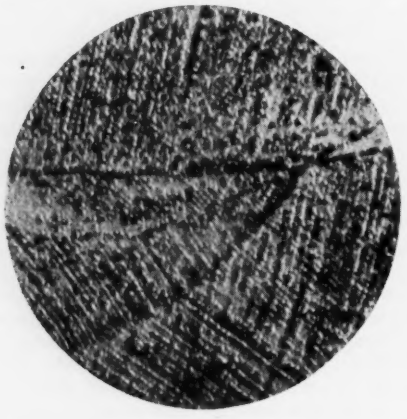

Fig. 11—Microstructure of steel in cold-drawn state, reheated to 650° C for 2 h × 1000

Fig. 12—Microstructure of steel in cold-drawn state, reheated to 760° C × 1000

Hence a new crystal structure is formed in specimens treated in this way. The surprising fact is that it appears and disappears in a very narrow temperature zone. It is probable that the corresponding phase, which denoted by *C*, exists in neighbouring systems, the 18/8 composition being at the boundary of its field of existence, and moreover is revealed only in samples subjected to preliminary plastic deformation.

Micrographic Study

To avoid lengthening the paper, the micrographic observations are summarized by emphasizing two points. No etchant satisfactorily reveals the α phase obtained by cold work. Subsequent heat-treatment does not alter this. On the other hand, it is difficult to account for globular carbide at temperatures above 550° C, unless it is assumed that this is an apparent effect due to an increase in the response to etching of the zones around each particle. The number of these globules decreases progressively during the $\alpha \rightarrow \gamma$ transformation, which corresponds, in all probability, to the dissolution of carbon in the γ phase (Figs. 11 and 12). The *C* phase could not be detected in the samples in which it was detected by X-rays.

Summary

Heat-treatment of 18/8 steels, partially transformed to martensite by cold work, brings about two important processes:

(i) A disappearance of the α phase, starting at 550° C, attaining great speed at 600–650° C, then slowing down considerably, and being completed at about 750° C

(ii) The abrupt precipitation of carbides at about 580° C, which progressively redissolve above this.

A reaction of secondary importance can occur in a limited zone around 580° C giving rise to the transient existence of a (previously) unknown phase *C*, revealed by X-rays and corresponding to a lattice not yet identified.

EFFECT OF HEAT-TREATMENT ON THE HARDNESS OF TRANSFORMED STEELS

The hardening induced in 18/8 steels by cold work is often considerable. What happens to this strain-hardening when such hardened steels are held at temperatures above the normal ? Measurements made on specimens quenched in water after holding for 2 h at various temperatures give an answer to this question.

Curve 1 of Fig. 13 relates to a steel containing 0·04% C, 17·81% Cr, and 9% Ni, cold-drawn to 77% reduction in area, and almost completely transformed to α phase. The hardnesses, measured after the quench, are approximately constant up to 500° C, but decrease rapidly beyond this.

Curve 2 of Fig. 13 relates to a steel partially transformed to the α state through 41·4% reduction in area : it contains 0·035% C, 18·3% Cr, and 10% Ni. Above 100° C the hardness increases considerably, reaches a maximum at 500° C, and then decreases to a value considerably below the initial one.

Curve 3 of Fig. 13 is presented to compare with the preceding case. The steel contains 0·035% C, 18·6% Cr, and 11·35% Ni, and was practically untransformed by 41·4% reduction in area. A little harder initially (probably because of its higher Ni content), this steel behaves like the preceding.

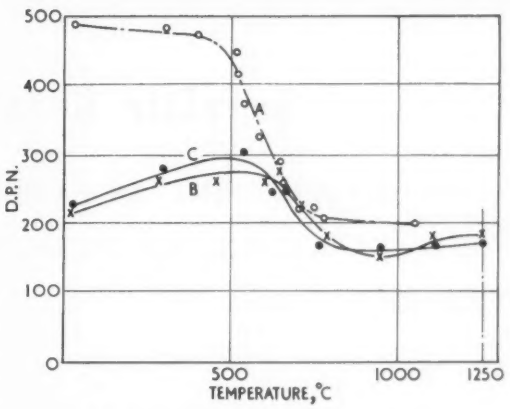

Fig. 13—(*a*) Variation of hardness measured by Vickers indentation under load of 5 kg after 2 h at temperatures indicated (18/8 steel cold-drawn 77% before treatment); (*b*) effect of heat-treatment on hardness specimens of a steel partially transformed by 41·4% reduction in area through a die; (*c*) effect of heat-treatment on hardness of specimens of another steel untransformed by 41·4% reduction in area through a die at room temperature

This shows that the increase in hardness observed in incompletely transformed steels is connected much more with the presence of γ than of α. However, its importance should not be overlooked, for it presents a means of increasing the hardness of an 18/8 steel, by a combination of a martensitic transformation of part of the austenite in the cold, and a structural hardening of the retained austenite.

DISCUSSION OF RESULTS

The above observations can act as points of departure for new studies of the effects of plastic straining on 18/8 steels of compositions chosen as being likely to yield combinations of mechanical properties that are frequently difficult to obtain unaccompanied by brittleness in other specially treated steels. To be worthy of attention, the new properties should not be gained at the expense of a considerable reduction of the corrosion resistance. It will be of interest, in the future, to undertake research into the exact field of application of 18/8 steels thus produced.

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'Bainitic Retained Austenite'

ITS 'CONDITIONING' IN En40C STEEL, AND ITS OCCURRENCE IN OTHER B.S. En STEELS

By J. A. Cameron, B.Sc.

SYNOPSIS

If En 40C steel is transformed by slow cooling through the bainite range, about 25% of retained austenite is obtained. The behaviour of this 'bainitic retained austenite' on tempering has been investigated by dilatometric methods, supplemented by hardness, X-ray data, microstructure, and microchemical analysis of electrolytically extracted carbide residues. It is shown that by suitable choice of tempering treatment the retained austenite present in the steel may be 'conditioned,' so that it transforms on cooling from the tempering temperature. The temperature at which this secondary transformation occurs on cooling appears to be a function of the carbon content of the retained austenite, which is affected by the temperature and duration of tempering. Conditioning is found to be accompanied by an increase in tensile strength. Several other En steels were examined for bainitic retained austenite after suitable treatment, and amounts up to 15% were found. 1150

IN A PREVIOUS PAPER[1] the occurrence of retained austenite formed by slow-cooling samples of En 40C steel through the bainite range was noted. The amount of retained austenite increased up to 25% as bainite formation progressed, although only half of this was formed by isothermal transformation. Since no retained austenite was obtained on water-quenching from the austenitizing temperature, the retained austenite obtained on slow cooling was thought to be associated with the bainite reaction and was termed 'bainitic retained austenite.' The carbon content of the retained austenite was deduced, from lattice parameter measurements, to increase to about 1·2% as bainite formation proceeded, although only 0·41% carbon was present in the parent austenite of the steel. In the theories of bainite formation,[2–4] it is not unanimous whether the reaction is one of nucleation and growth or of the martensitic type; but it seems accepted that carbon diffusion from the freshly formed ferrite into the surrounding austenite does occur. The formation of bainitic retained austenite is in accordance with this aspect of the theories to such an extent that the surrounding austenite became progressively enriched in carbon as increasing amounts of bainite were formed, until the stability of the austenite was such that it was retained at room temperature. It was suggested that in a suitable tempering treatment, this retained austenite may be 'conditioned,' so that part of it transformed on cooling from the tempering temperature.

The term 'conditioning' has been used previously with reference to the retained austenite occurring in high-speed steels,[5] and it is believed that, on tempering such steels, precipitation of carbides from the austenite occurs which decreases the stability of the retained austenite and so results in its decomposition on cooling from the tempering temperature. The practice of double and treble tempering of high-speed steel tools is associated with 'conditioning' of retained austenite.

The present investigation is concerned mainly with a dilatometric study of the effects of temperature and time on the decomposition of bainitic retained austenite in En 40C steel; it also deals with the effect of conditioning on mechanical properties, and the occurrence of bainitic retained austenite in several other B.S. En steels.

Manuscript received 15th April, 1955.

Mr. Cameron, who was formerly at the Brown-Firth Research Laboratories, where this work was carried out, is now at the Nelson Laboratories of the English Electric Co., Ltd., Stafford.

STEEL USED AND EXPERIMENTAL METHODS

Throughout this work, the En 40C material used was from the same heat of basic electric arc steel as that in the previous work[1] and had the following analysis:

C, %	Si, %	Mn, %	S, %	P, %
0·41	0·29	0·63	0·006	0·011

Ni, %	Cr, %	Mo, %	V, %
0·24	3·43	0·97	0·20

The austenitizing treatment was for 30 min at 940° C, and the austenitic grain size was A.S.T.M. 8. For the dilatometer tests, the specimen used was 0·335 in. dia. with an effective length of 4·0 in. Figure 1 shows the specimen assembled in the dial-gauge dilatometer.

For treatment before the dilatometer tests, the specimens were inserted in hollow heat-resisting steel cylinders (of 1 in. external dia. and $\frac{1}{4}$ in. wall thickness, with screwed plugs at each end), and after austenitizing, they were cooled in the cylinders in air (no pearlitic transformation) to 550° C, then transferred to a furnace at 550° C and cooled at a linear rate of 5° C/h down to the required temperature and water-quenched. By heat treating the specimens

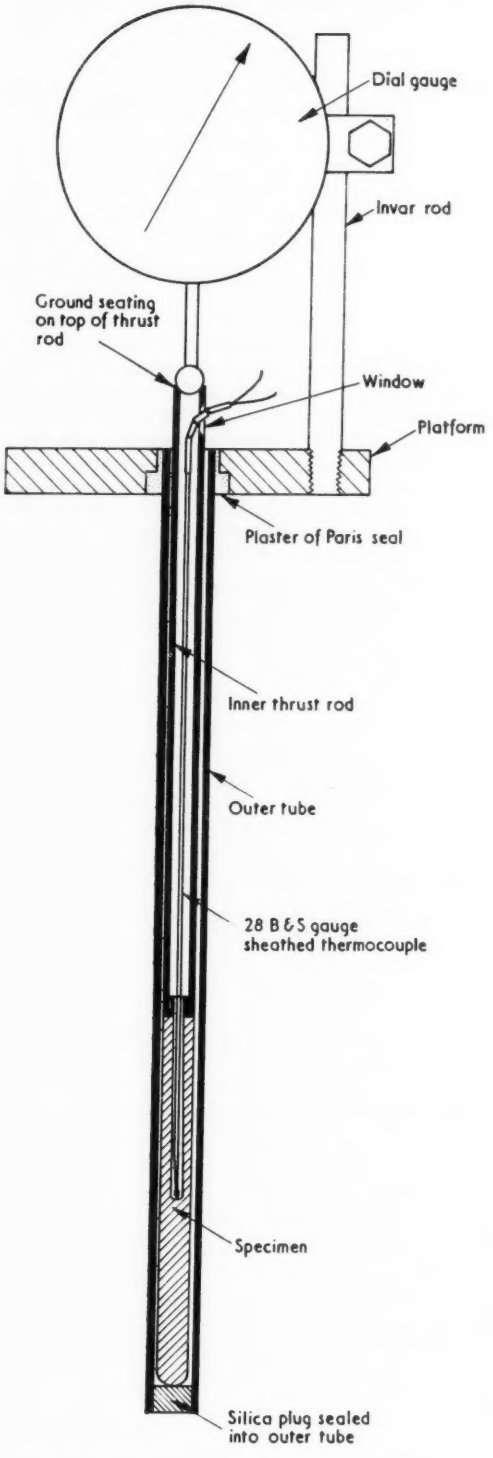

Fig. 1—Specimen assembled in dilatometer

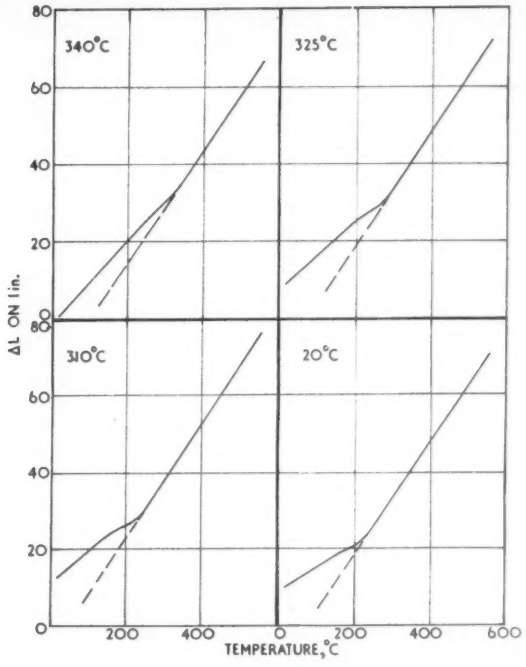

Fig. 2—Dilatation results, series *A*

inside closed cylinders, surface scaling was avoided. After treatment, the specimens were tested for hardness, and the amount of retained austenite was determined by X-rays. The X-ray estimates were obtained as before,[1] and are believed to be correct to the nearest 5%.

A lead bath was used for the dilatometer tests and its temperature was controlled to within $\pm 2°$ C of the required holding temperatures. The time taken for the dilatation test piece to reach the temperature was 15 minutes.

DILATOMETER TESTS

Series A

The first series of dilatation tests was done to determine the stability of bainitic retained austenite on tempering, as influenced by the temperature at which bainite formation was arrested during cooling at 5° C/h.

Four dilatometer specimens were austenitized and cooled in the cylinders to 550° C followed by cooling from 550° C at 5° C/h. During cooling, specimens 1, 2, and 3 were water-quenched at 340°, 325°, and 310° C respectively, whilst specimen 4 was allowed to continue cooling at 5° C/h down to 20° C. The amount of retained austenite was estimated by X-rays and the hardness was measured.

Each specimen was assembled in the dial-gauge dilatometer, placed in a lead bath at 555° C, and held at that temperature for $2\frac{3}{4}$ h. The dilatation/temperature curve on heating was obtained and the dial-gauge readings were noted during the soak at 555° C. The dilatometer was then removed from the lead bath and allowed to cool in air to room temperature.

Table I
EFFECTS OF DILATATION TESTS

Specimen	Treatment, 5° C/h to	Before Tempering		After Tempering		Temp., °C from Fig. 2
		D.P.N/ 30 kg	% γ	D.P.N/ 30 kg	% γ	
1	340° C, W.Q.	549	15–20	504	†	320
2	325° C, W.Q.	482	25	520	5	280
3	310° C, W.Q.	465	25	538	5	250
4	20° C, W.Q.	465	25	532	5	245

† Not detected—with the method used, less than 2–3% was not detectable

Table II
TESTS AT 555° C

Specimen	Time held at 555° C, h	After Test		Temp. T, °C
		D.P.N./30 kg	Retained Austenite, %	
5	$\frac{1}{4}$	524	10–15	185
3	$2\frac{3}{4}$	538	5	250
6	22	518	5	340
7	70	433	†	400

† Not detected

During cooling, the dilatation/temperature curve was obtained. The specimens were tested for hardness and by X-ray methods for retained austenite. Figure 2 shows the dilatation curves on these specimens on cooling. No dilatation changes were observed during the holding period at 555° C, but during cooling a change of gradient was observed in the dilatation curves. The temperature at which this change occurred (subsequently referred to as T), together with hardness and retained austenite contents before and after the dilatation tests, is given in Table I.

The hardness differences in Table I before tempering were due to the incomplete transformation to bainite down to 310° C with consequent differences in the amount of martensite formed on quenching. The decrease in hardness of specimen 1 after the 555° C treatment was probably caused by the tempering of the martensite, more of which was present in specimen 1 than in the other specimens. The retained austenite was reduced considerably by treating at 555° C, and the hardness of specimens 2, 3, and 4 was increased by 8–16%. The dilatation changes observed are consistent with the transformation of retained austenite on cooling from 555° C; the hardness and X-ray data support this interpretation. The temperature at which transformation started decreased from 320° C for specimen 1 to 250° C for specimen 3; this aspect of the results will be discussed later.

Series B

The second series of dilatometer tests was carried out to investigate the effect of tempering temperature and time on the transformation of bainitic retained austenite.

The tests in this series were done on specimens austenitized and cylinder-cooled to 550° C then cooled at 5° C/h to 310° C followed by water-quenching. This treatment gave a hardness of 465 D.P.N. and 25% retained austenite.

At 555° C

Dilatation specimens 5, 3, 6, and 7 were held at 555° C for $\frac{1}{4}$, $2\frac{3}{4}$, 22, and 70 h respectively and the dilatometer cooled in air as before. The dilatation curves obtained are shown in Fig. 3; the hardness and retained austenite contents after the test, together with T the transformation temperature, (in ° C) of the retained austenite on cooling from 555° C, are given in Table II.

From the data in Table II it appeared that $\frac{1}{4}$ h at 555° C resulted in the transformation (during cooling) of only one-half of the retained austenite present before the treatment, whereas holding at this temperature for longer times substantially reduced the amount of retained austenite of specimens 3, 6, and 7. The hardness after the dilatation tests was appreciably increased from the initial 465 D.P.N. for holding times from $\frac{1}{4}$ h to 22 h, and slightly decreased for a holding time of 70 h. In these tests there was no dilatometric evidence of changes occurring at 555° C, but on cooling from 555° C, T increased progressively from 185° C to 400° C for holding times of $\frac{1}{4}$ and 70 h respectively.

At 460° C

Dilatation specimens 8, 9, and 10 were held at 460° C for $2\frac{3}{4}$, 22, and 70 h, respectively. The dilata-

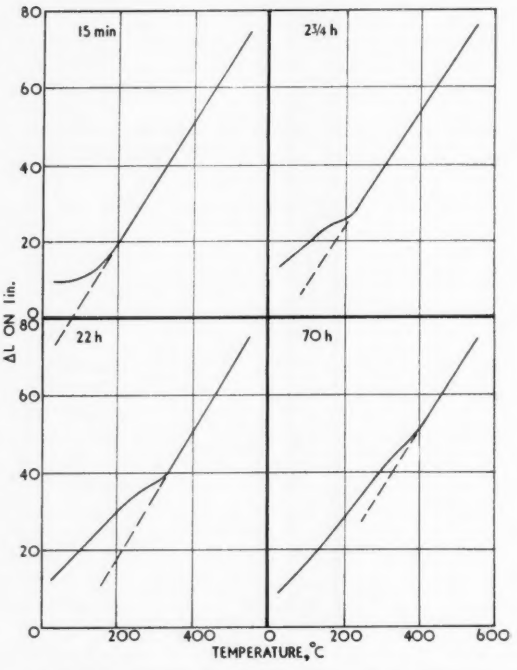

Fig. 3—Dilatation results, series *B* at 555° C

Table III
TESTS AT 460° C

Specimen	Time held at 460° C, h	After Test		Temp. T, ° C
		D.P.N./ 30 kg	Retained Austenite, %	
8	$2\frac{3}{4}$	472	20	Not observed
9	22	508	10–15	120
10	70	526	10	155

Table IV
TESTS AT 635° C

Specimen	Time held at 635° C, h	After Test		Temp. T, ° C
		D.P.N./ 30 kg	Retained Austenite, %	
11	$\frac{1}{4}$	478	†	320
12	$2\frac{3}{4}$	385	†	430
13	22	319	†	Not observed

† Not detected

tion curves obtained are shown in Fig. 4, and the other details in Table III.

The stability of the retained austenite at 460° C was much greater than at 555° C. In specimen 8, held $2\frac{3}{4}$ h at 460° C, only 5% retained austenite was removed and no transformation of austenite was detected dilatometrically on cooling. In Test 9, consisting of holding 22 h at 460° C, half of the retained austenite was removed, and there was evidence of transformation starting at 120° C on cooling. After 70 h at 460° C, 10% retained austenite remained, and that portion which transformed on cooling began transformation at 155° C, whilst the hardness increased from 465 to 526 D.P.N. As for conditioning at 555° C, the temperature at which transformation started was increased for longer holding times, but this temperature was much lower for the 460° C series.

At 635° C

Specimens 11, 12, and 13 were held at 635° C for $\frac{1}{4}$, $2\frac{3}{4}$, and 22 h respectively. The results are given in Table IV, and the dilatation curves are shown in Fig. 5.

At 635° C the stability of the retained austenite was much reduced. For the specimen held $\frac{1}{4}$ h at 635° C, there was no dilatometric evidence of transformation at temperature. The decomposition of the retained austenite started at 320° C on cooling, and the hardness after the test was slightly higher than before. The specimen held $2\frac{3}{4}$ h at 635° C was observed to expand slightly after 45 min, presumably because of the beginning of the isothermal breakdown of the retained austenite to ferrite and carbide. The amount of isothermal transformation at 635° C, after $2\frac{3}{4}$ h, as indicated by the dial gauge, was, however, quite small; the transformation of the remaining austenite started at 430° C on cooling and was complete at the end of the test. The hardness decreased to 385 D.P.N. In the case of specimen 13, which was held 22 h at 635° C, isothermal transformation was observed to begin after 45 min, and continued during the period of holding at temperature. From the dilatometer curves, it was thought that most of the retained austenite transformed isothermally at 635° C. The hardness decrease observed, resulted probably from the tempering of the bainite, the decomposition products of retained austenite at 635° C being relatively soft.

Series C

This work was initially undertaken as a practical investigation relating to the normalizing of large forgings, which are reheated for tempering without being allowed to become cold. Since the treatment was interrupted by water-quenching from 310° C, it was considered necessary to determine whether the conditioning of the retained austenite would be similar if the specimen were immediately reheated from 310° C, instead of water-quenching from 310° C before tempering.

Specimen 14 was placed inside the dilatometer, and the open end of the dilatometer tube was plugged with asbestos wool. The specimen was then austenitized in the dilatometer (with the top platform outside the furnace), then cooled in air to 550° C, followed by cooling at 5° C/h to 310° C. The inner thrust rod and thermocouple were inserted, and the whole quickly transferred to a lead bath at 500° C. The dial gauge was fixed in position and initial readings taken before heating the lead bath to 555° C. The specimen was held at that temperature for $2\frac{3}{4}$ h and air-cooled in the dilatometer. An arbitrary zero reading at 20° C was chosen, and the dilatometer curve is shown in Fig. 6*a*. After the test, the hardness measured on this specimen was 549 D.P.N. and the retained austenite content was found by X-rays to be 5%. The temperature T from Fig. 6*a* was 230° C, i.e. essentially the same as for specimen 3, the

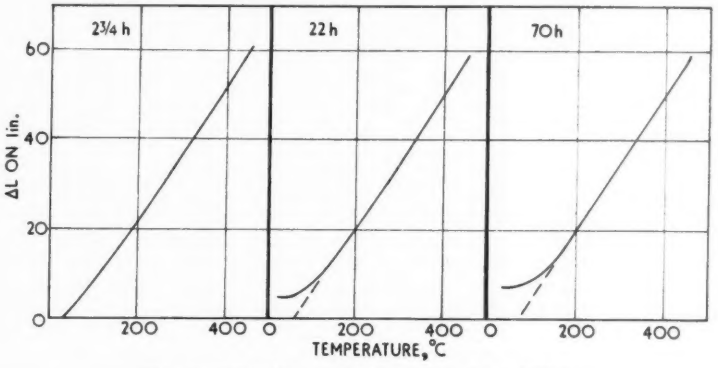

Fig. 4—Dilatation results, series *B* at 460° C

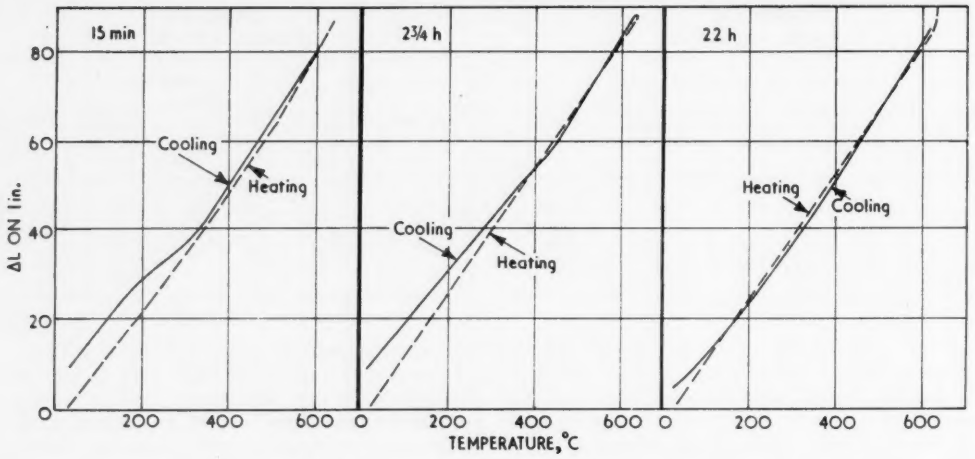

Fig. 5—Dilatation results, series *B* at 635° C

treatment of which differed from Specimen 14 in that the former was water-quenched from 310° C.

As a control test, a dilatometer specimen in the condition 30 min, 940° C, O.Q. (no retained austenite) was tested. This specimen (15) was held $2\frac{3}{4}$ h at 555° C and air-cooled. The dilatation curves obtained are shown in Fig. 6*b*. The hardness of Specimen 15 before and after testing was 680 and 515 D.P.N. respectively; no changes of the type attributed to conditioning were observed.

Carbide Residues

Carbide residues were obtained from selected dilatometer specimens (also specimens *X*, *Y*, and *Z* in Table V) by electrolytic extraction,[6] and these were analysed by micro-analytical methods for iron, chromium, molybdenum, and vanadium (*see* Table V). On the left, the percentage of each of these elements present in the residue is expressed as a percentage of that element in the original steel. The total in the column on the left, for each specimen, indicates a measure of the residue yield. The percentage of each element in the total is given in the column on the right.

The lowest residue yield values were given by *X* and *Y*. In *X*, which was oil-quenched from 940° C, the carbide residue contained only the out-of-solution carbides in the austenitizing treatment; the residue was very rich in molybdenum and vanadium and was low in iron. The residue from sample *Y*, which was water-quenched from 310° C, was also rich in molybdenum and vanadium.

For specimens in the condition 310° C, W.Q., the effect of tempering at 555° C on the residue yield is shown, by comparing the totals of specimens *Y*, 3, and 7, to increase the amount of residue in the ratio 1 : 3 : 9 for no tempering, $2\frac{3}{4}$ h at 555° C, and 70 h at 555° C respectively. A comparison of the results for specimens 3 and *Z* showed that on water-quenching from the tempering temperature (as opposed to air cooling), the residue yield was decreased. This suggested some precipitation of carbides on cooling from the tempering temperature.

Microstructure

In Fig. 7 is shown the microstructure of specimen *Y* which was treated for 30 min at 940° C, air-cooled to 550° C, then cooled at 5° C/h to 310° C and water-quenched. This sample contained ferrite (bainite which is almost carbon free), martensite, and 25%

Table V
MICRO-ANALYSES OF CARBIDE RESIDUES

Specimen	*X*		15		*Y*		3		*Z*		7	
Treatment* prior to extraction	*B*		*B* + $2\frac{3}{4}$ h, 555° C, W.Q.		*A*		*A* + $2\frac{3}{4}$ h, 555° C, A.C.		*A* + $2\frac{3}{4}$ h, 555° C, W.Q.		*A* + 70 h, 555° C	
Fe	0·09	26	0·68	53	0·11	30	0·63	53	0·38	54	2·07	57
Cr	0·04	12	0·41	32	0·11	30	0·30	25	0·17	24	1·12	31
Mo	0·15	44	0·10	8	0·08	22	0·15	12·5	0·11	15·5	0·26	7
V	0·06	18	0·09	7	0·07	19	0·10	8·5	0·05	7·0	0·15	4
Total	0·34		1·28		0·37		1·18		0·71		3·60	

* Treatments: *A* 30 min, 940° C, A.C. to 550° C, 5° C/h to 310° C, W.Q.
B 30 min, 940° C, O.Q.

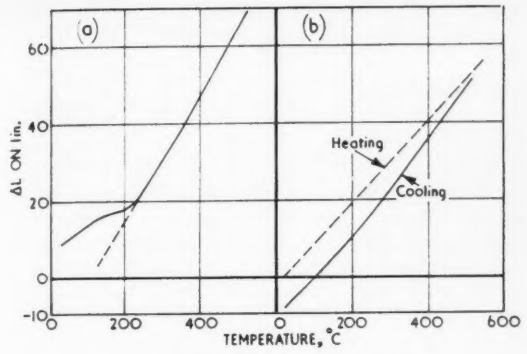

Fig. 6—Dilatation results, series *C*

retained austenite. The latter could not be detected microscopically.

An aid to the metallographic interpretation in this work was obtained by comparing the microstructure of specimen *Y* with those of specimens 3 and 14 shown in Figs. 8 and 9. The latter specimens were cooled from 550° C at 5° C/h to 310° C, from which temperature specimen 3 was water-quenched, then treated for $2\frac{3}{4}$ h at 555° C and air-cooled; whereas, for specimen 14, the water-quench from 310° C was omitted and the specimen was heated from 310° to 555° C, held $2\frac{3}{4}$ h, and air-cooled. Specimen 3 showed black etching patches which were absent in specimens *Y* and 14. From the difference in heat treatment it was inferred that the black etching areas were (tempered) martensite. This martensite was formed from some of the austenite untransformed on reaching 310° C; the remaining untransformed austenite was, of course, retained at room temperature. Apart from the black etching patches, the microstructure of samples 3 and 14 appeared to be essentially similar, namely, a matrix of ferrite with carbide particles distributed throughout (specimen 14 contained untempered martensite). The effect of holding 70 h at 555° C (as in specimen 7) on the amount of carbide precipitation is illustrated in Fig. 10.

Fig. 7—Specimen 7: ½ h at 940° C, A.C. to 550° C, 5° C/h to 310° C, W.Q. Etched 2% nital × 1500

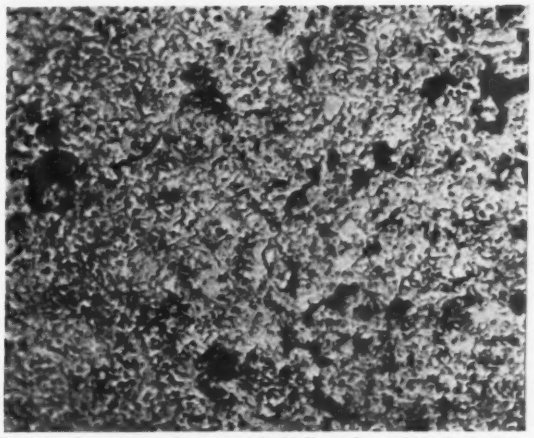

Fig. 8—Specimen 3: ½ h at 940° C, A.C. to 550° C, 5° C/h to 310° C, W.Q. + $2\frac{3}{4}$ h 555° C, A.C. Etched 2% nital × 1500

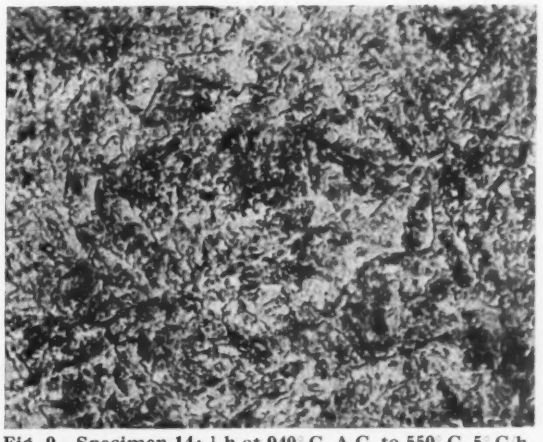

Fig. 9—Specimen 14: ½ h at 940° C, A.C. to 550° C, 5° C/h to 310° C, W.Q. + $2\frac{3}{4}$ h 555° C, A.C. Etched 2% nital × 1500

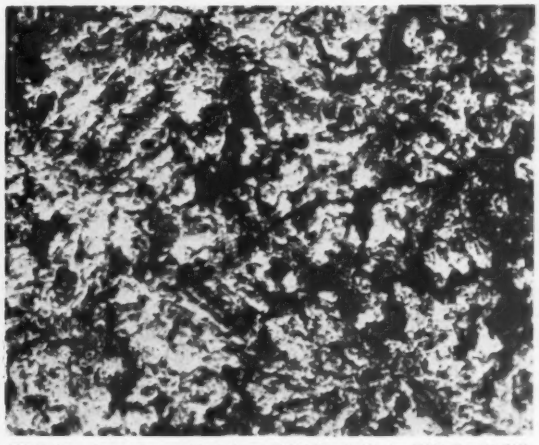

Fig. 10—Specimen 7: ½ h at 940° C, A.C. to 550° C, 5° C/h to 310° C, W.Q. + 70 h 555° C, A.C. Etched 2% nital × 1500

Table VI

DETAILS OF TREATMENT AND TENSILE RESULTS

Specimen	Treatment*	L.P.	Proof Stress, tons/in²			Max. Stress, tons/in²	Elongation, %	Reduction in Area, %
			0·05%	0·1%	0·5%			
A	W.Q.	15	31	37	60	105	17	29
B	W.Q. + 2¾ h, 555° C furnace cool	30	49·5	58·5	85·5	114	15	41
C	Omit W.Q. and reheat to 555° C, hold 2¾ h, furnace cool	26·8	46	55	82	121	15	29
D	As for *C* + 2¾ h, 555° C furnace cool	40	65·5	74·5	97·5	115	15	38

* All specimens were treated for ½ h at 940° C, cylinder-cooled to 550° C, then cooled at 5° C/h to 310° C before the treatment indicated

Tensile Tests

A few tests were carried out to find the effect of conditioning on the tensile properties of this steel. The finished, machined specimens were treated inside heat-resisting steel cylinders. The treatment details and tensile results are given in Table VI.

These results showed that transformation of the retained austenite following conditioning was accompanied by increased limit of proportionality, proof stress, and maximum stress; and that, in a second treatment at 555° C, the limit of proportionality and proof stress were further increased, while the maximum stress decreased to a value which was, however, greater than that before conditioning. The limit of proportionality before conditioning was exceptionally low, probably owing to premature plastic deformation associated with the retained austenite.

The tensile strength of specimen *C* was highest as it contained no tempered martensite (*see* section on microstructure).

Bainitic Retained Austenite in other B.S. En Steels

For academic interest, several B.S. En steels were examined for bainitic retained austenite. No retained austenite was detected by X-ray methods when these steels were water-quenched from 835° C, but when treated for ½ h at 835° C, and air-cooled to 500° C, followed by cooling at 5° C/h to 20° C, some retained austenite was found. A list of the steels tested, together with hardness and X-ray data, is given in Table VII.

As treated, all these steels contained some retained austenite. Apart from En 40B, the greatest quantity, 15%, was found in En 30B. There may be a very approximate relationship between the amount of retained austenite as treated and the total chromium and molybdenum content of the steel. After austenitizing for ½ h at 835° C, samples of En 30B and En 25 were isothermally treated for 20 h at 400° and 350° C, but in neither steel was retained austenite detected. A common feature of the steels examined is that, like En 40C, their isothermal transformation diagrams[7] showed incomplete transformation in the upper bainite region.

DISCUSSION

The similarity of the residue yields in samples *X* (O.Q., 940° C) and *Y* (W.Q., 310° C) indicates that the residue obtained from *Y* is mainly carbide which was out of solution at the austenitizing temperature. This is consistent with the deduction that sample *Y* consists of carbon-free ferrite, about 10% of martensite, and 25% retained austenite, with no precipitated carbide. Specimen *Z* was given the same treatment as *Y* followed by tempering for 2¾ h at 555° C,

Table VII

BAINITIC RETAINED AUSTENITE IN OTHER STEELS

B.S. En No.	Analysis, %						As-treated Hardness, D.P.N.	γ by X-ray, %
	C	Si	Mn	Ni	Cr	Mo		
23	0·25	0·23	0·59	3·46	0·78	...	287	<5
24	0·37	0·24	0·66	1·64	1·20	0·29	321	5–10
25	0·29	0·28	0·58	2·69	0·76	0·57	317	10
26	0·38	0·21	0·58	2·51	0·66	0·44	305	<5
30A	0·29	0·27	0·43	4·20	1·30	0·08	340	10
30B	0·29	0·27	0·49	4·13	1·28	0·32	373	15
40B	0·28	0·25	0·56	0·20	3·26	0·51	393	15–20
100	0·46	0·27	1·37	0·79	0·52	0·24	343	10
110	0·41	0·23	0·66	1·38	1·20	0·18	309	<5

from which temperature it was water-quenched. The residue yield in *Z* has increased by 0·71–0·37 = 0·34. Some of this increase of 0·34 may be associated with carbide precipitation during tempering, from the 10% martensite present. However, the comparison between samples *X* (O.Q., 940° C, fully martensitic) and 15 (O.Q., 940° C + 2¾ h at 555° C) indicates an increase of 1·28–0·34 = 0·94 in residue yield for 100% martensite, i.e. an increase of about 0·09 for 10% martensite. The larger part of the increased residue yield in sample *Z* may, therefore, be attributed to carbide precipitation from the austenite. This loss of carbon from the retained austenite rendered it less stable (i.e. ' conditioned ' it), so that transformation occurred during cooling from the tempering temperature thus increasing the hardness. For specimen 3, the temperature at which transformation began on cooling was 250° C. This temperature suggested that the transformation of the retained austenite on cooling from 555° C was to martensite, although the comparison of the residue yields of specimens 3 and *Z* indicated that some carbide was precipitated on air-cooling from 555° C. However, whatever the transformation product on cooling from 555° C, the temperature of the beginning of transformation *T* would be primarily influenced by the carbon content of the retained austenite at the end of the tempering treatment, i.e. the temperature *T* would be lower the higher the carbon content.

In the case of specimen 7, which was conditioned for 70 h at 555° C, the residue yield was very high, the microstructure showed much carbide precipitation, and the temperature of the beginning of transformation on cooling was 400° C.

Considering the other tempering times used (*see* Table II), tempering at 555° C for 22 h gave a temperature *T* of 340° C, whereas for specimen 5 which was held ¼ h at 555° C, the carbide precipitation effected conditioning of only half the retained austenite present, and that portion which was conditioned had a temperature *T* of 185° C.

The carbon content of the retained austenite at 310° C was deduced from lattice parameter measurements[1] to be 1·2%. Similar determinations on specimen 5, which contained 10–15% retained austenite after conditioning for ¼ h at 555° C, indicated a carbon content of 0·60%.

Therefore it is evident that for conditioning at 555° C the temperature of the beginning of transformation on cooling was a function of the carbon content of the retained austenite at the end of the conditioning period, and that temperature *T* increased with the conditioning time.

For conditioning at 460° C, the diffusion rate of the carbon would be much lower, and even after 70 h at 460° C, 10% retained austenite remained: the temperature *T* was 155° as opposed to 185° C for conditioning for ¼ h at 555° C.

The specimens tempered at 635° C expanded after 45 min at temperature, indicating the beginning of the breakdown of the austenite isothermally. Specimen 3, which was held for ¼ h at 635° C was, however, conditioned, the temperature *T* being 320° C. That portion of the retained austenite which resisted isothermal transformation at 635° C began to transform at 430° C on cooling.

The differences in the values of *T* in Table I are again due to differences in the carbon content of the retained austenite; for it has been shown[1] that on slow cooling through the bainite range, the carbon content of the austenite increased from 1·0% at 340° C to 1·2% at 310° C and 20° C. Although specimens 1–4 were given identical conditioning treatments, the carbon content of the austenite before tempering determined the difference at the end of the conditioning treatment, hence the values of *T* increased as the starting temperature decreased to 310° C.

CONCLUSIONS

(1) It has been shown that retained austenite formed by cooling En 40C steel slowly through the bainite range may be conditioned in tempering, so that transformation occurs on cooling from the tempering temperature.

(2) The temperature at which transformation begins on cooling from the tempering temperature depends on the carbon content of the retained austenite at the end of the conditioning treatment.

(3) The transformation of retained austenite after conditioning increases the tensile strength of the steel; the limit of proportionality is low before conditioning.

(4) Several other B.S. En steels form bainitic retained austenite on cooling at 5° C/h below 500° C. Of the steels examined, the most notable was En 30B which contained 15% retained austenite.

Acknowledgments

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Some Experiments on the Composition of Carbides in Low-alloy Steels

By J. E. Bowers, B.Met., Ph.D., A.I.M.

SYNOPSIS

Variations in carbide composition in a series of six low-alloy steels have been investigated as a function of temperature of isothermal transformation, using X-ray and chemical methods.

The iron carbides formed in the bainite reaction contained only a small proportion of the carbide-forming elements added. Some segregation of these elements into the carbide does occur, especially in the upper bainite range.

In steels with small manganese additions the amount of manganese in the carbide increased progressively as the temperature was raised within the pearlite range. In steels of higher alloy content (e.g. steel *CR*4 which contained 3% of chromium), the carbide resulting from the pearlite reaction was the same at all temperatures. This constant value was not the equilibrium composition in the two 3% chromium steels studied.

Changes in the carbide phase during tempering were studied on the 3% chromium steels *CR*4 (0·5% C) and *CR*5 (0·2% C). Chromium diffused into the carbide phase until the equilibrium composition was attained; the equilibrium was established more rapidly at higher temperatures. The mechanism involved in the transformation of Fe_3C to Cr_7C_3 is uncertain. 1270

SINCE THE PROPERTIES of a steel depend upon the composition and distribution of the phases present, a considerable amount of work has been done on the carbides formed during various heat treatments. Good reviews of this subject are given in several papers[2, 11] and only a brief note on some of the relevant work will be given here.

By X-ray analysis it has been shown that the metal atoms in a carbide can be replaced to a limited extent by atoms of other carbide-forming elements, without changing the crystal structure. Westgren, Phragmén, and Negresco,[1] for instance, found that up to 55% of the chromium in the trigonal carbide Cr_7C_3* could be replaced by iron.

Several investigations have shown that the carbides formed during isothermal transformation are not the same at all temperatures.[2, 3, 4, 10, 13, 14, 16] The bainite reaction produces an iron carbide of low alloy content, and Hultgren[3] has shown that for several low-alloy steels the proportions of alloying elements to iron in the carbide were the same as in the parent austenite. During the early stages of the pearlite reaction, cementite of low alloy content is precipitated, but as the transformation proceeds the amount of alloying element in the carbide increases. Similarly, during the tempering of a quenched alloy steel, the carbide-forming elements present segregate into the carbide phase.[5, 6] If the proportion of alloying element to carbon is sufficiently high, an alloy carbide is formed during the later stages of the pearlite reaction or after prolonged tempering at a high subcritical temperature.

An attempt has been made in the present investigation to study the effect of temperature of isothermal transformation on the carbides produced in a series of six low-alloy steels. The influence of both time and temperature of tempering have been studied on the two 3% chromium steels *CR*4 (0·5% C) and *CR*5 (0·2% C).

EXPERIMENTAL PROCEDURE

The isothermal transformation curve for each steel was first determined, and, from the transformation data thus obtained, the time needed to reach the end of the transformation process was ascertained for the temperatures required. The samples from which carbides were to be extracted for analysis were transformed for this predetermined time and then machined to remove any decarburized surface. Carbides obtained from these samples by anodic solution were analysed by chemical and X-ray methods. All the results given have been obtained from analyses on at least two separately extracted residues. A 9-cm powder camera was used for the X-ray analysis.

A number of bars of the 3% chromium steels *CR*4 and *CR*5 were austenitized by soaking at 950° C for ½ h, and then oil-quenched. These bars were subsequently tempered for various times at temperatures of 400–700° C. Any decarburized surface layer was removed by pickling in hydrochloric acid. The carbides were extracted from these samples and analysed, using the same technique as that employed in the experiments on isothermal transformation.

The chemical compositions of the materials used are given in Table I.

Determination of Isothermal Transformation Curves

A dilatometric method was used for determining the isothermal transformation curves. The sample

*The trigonal carbide will henceforth be given as Cr_7C_3, it being understood that this does not represent the chemical composition of the carbide. Similarly, other carbides will be given as Fe_3C, etc., to indicate the parent structure.

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Table I
ANALYSES OF STEELS USED

Steel	C, %	Si, %	Mn, %	P, %	S, %	Ni, %	Cr, %	Mo, %
*MS*1	0·33	0·28	1·63	0·015	0·013	...	0·17	...
FA	0·29	0·20	1·45	0·019	0·024	...	...	0·26
*CR*8	0·36	0·19	0·78	0·078	0·030	0·33	0·97	0·06
*MO*2	0·45	0·19	0·47	0·025	0·029	0·32	0·31	2·42
*CR*4	0·51	0·20	0·38	0·014	0·013	0·06	2·97	...
*CR*5	0·22	0·17	0·25	0·010	0·012	0·06	3·03	...

under test was austenitized for ½ h at 950° C in an atmosphere of nitrogen and then quenched into a lead bath which was controlled to within ± 2° C of the desired transformation temperature. A dial gauge indicated the change in length of the sample during the transformation. The hardness and microstructure at various stages during the transformation gave a useful check on the dilatometric measurements. The only exception to this procedure was the molybdenum steel *MO*2 which was austenitized at 1050° C for 4 h to take the alloy carbide into solution.

Separation of Carbides

Various solvents have been used for dissolving away the ferrite matrix of a steel whilst leaving the carbides untouched. Direct chemical attack of the sample is a very slow process and better results are obtained when the sample is decomposed by making it the anode in an electrolytic cell. When separating the carbide from a plain carbon steel, a neutral electrolyte gives less decomposition of the carbide than an acid solution. On the other hand, many of the carbides formed in alloy steels can be separated using an acid electrolyte, whilst with a neutral electrolyte some of the ferrite would remain undissolved. These steels contain only small amounts of inclusions which could be left behind in the residue.[7]

A 5% aqueous solution of hydrochloric acid was used as an electrolyte in the present series of experiments and a piece of platinum foil served as a cathode. After dissolving away the anode, the carbide was filtered off and washed, first with 5% hydrochloric acid and then with distilled water. The residue was dried by desiccation over calcium chloride.

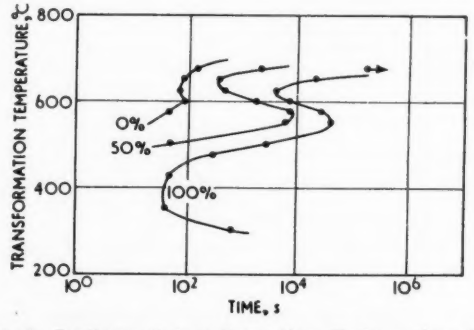

Fig. 1—Isothermal transformation diagram for steel *FA* (0·29% C; 1·45% Mn; 0·26% Mo) as determined by dilatometric measurements

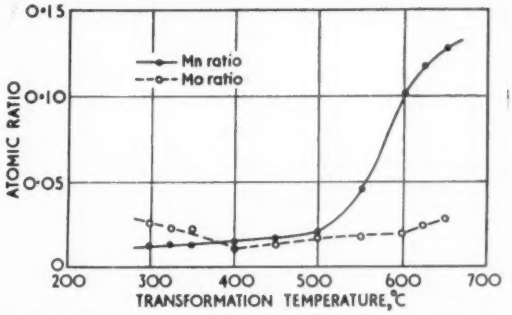

Fig. 2—Change in composition of the carbide phase with the variation in temperature of isothermal transformation for steel *FA* (0·29% C; 1·45% Mn; 0·26% Mo). Manganese ratio = Mn/Fe + Mn + Mo; molybdenum ratio = Mo/Fe + Mn + Mo

Presentation of the Chemical Analyses

The residue obtained is not necessarily a pure carbide. Using an acid electrolyte on the low-alloy steels selected for the present experiments prevents contamination of the residue with undissolved ferrite or with insoluble basic salts from the electrolyte. It is unwise to rely on the chemical analysis when estimating the carbon content of the carbide, since during separation some decomposition may occur, leaving carbon in the residue. It is better to calculate the carbon content of the carbide from the structure as determined by X-ray analysis. As the present study has been concerned mainly with the changes in alloy content of the carbide with varying heat-treatment, the carbon contents of the carbides have not been calculated. Instead the results have been reported as the number of atoms of the element concerned to the total number of metal atoms in the carbide; for example, the manganese content of a carbide extracted from the steel *FA* is expressed as the ratio of manganese atoms to manganese + iron atoms, there being only these two carbide-forming elements in the residue.

EXPERIMENTAL RESULTS

Manganese Steels *MS*1 and *FA*

These steels contain 1½% of manganese and 0·3% of carbon; the steel *FA* also has ¼% of molybdenum added. The transformation diagrams for these steels (Figs. 1 and 3) show no unusual features.

In Fig. 4 the manganese content of carbides extracted from completely transformed samples of steel *MS*1 is plotted against the temperature of transformation. Below 450° C the manganese content of the carbide was low and the variation with temperature was slight; this is what one would expect in view of the short times required for complete transformation of these samples. In the upper bainite range the increase in time required for complete transformation allows more diffusion of the alloying element into the carbide phase. Thus at 450° and 500° C some segregation of manganese into the carbide was detected. At 550–600° C the product was pearlite with even more manganese in the carbide, the increase in mobility of the manganese atoms at the higher temperatures more than compensating for the shorter

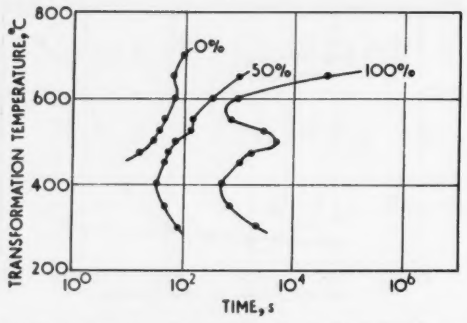

Fig. 3—Isothermal transformation diagram for steel *MS*1 (0·33% C; 1·63% Mn)

transformation times. The manganese content of the carbide rises to its maximum value at the highest temperature studied (650° C). The carbides all had the orthorhombic Fe_3C structure.

The carbide-forming elements present in steel *FA* are iron, manganese, and molybdenum; hence the composition of the carbide phase (*see* Fig. 2) has been expressed as the ratio of these atoms in the carbide, i.e. Mn/Fe + Mn + Mo and Mo/Fe + Mn + Mo indicate the manganese and molybdenum contents respectively. At temperatures below 550° C the transformation product is bainite and the manganese content of the carbide is very low. Within the pearlite range the manganese content of the carbide phase increases as the transformation temperature is raised. The molybdenum content of the carbide phase is constant within the limits of accuracy of the separation and analytical methods used. At all temperatures the carbide was Fe_3C with a small proportion of the iron replaced by manganese and molybdenum.

It is interesting to note that Wever and Mathieu,[8] from an investigation of the Curie point of carbides in a manganese steel, concluded that the manganese content of the carbide decreased as the transformation temperature was lowered.

Steel *CR*8 (0·36% C; 0·97% Cr; 0·78% Mn)

Carbides separated from completely transformed samples of this steel were all of the form Fe_3C, some of the iron being replaced by chromium and manganese. The ratios of metallic elements in the carbides are plotted against transformation temperature in Fig. 6. At low temperatures, below about 450° C, almost pure cementite is formed. The amount of chromium and manganese in the carbide increases as the transformation temperature is raised above 450° C and rises to an approximately constant value above 550° C. The ' nose ' of the isothermal transformation curve occurs at about 550° C (*see* Fig. 5).

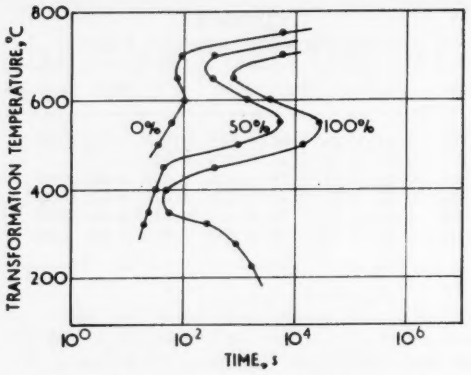

Fig. 5—Isothermal transformation diagram for steel *CR*8 (0·36% C; 0·97% Cr; 0·78% Mn)

Steel *MO*2 (0·45% C; 2·42% Mo)

This steel was austenitized at 1050° C to ensure complete solution of the alloy carbide. The ($\gamma \rightarrow \alpha$) transformation was very sluggish and in the temperature range 450–600° C no results have been obtained, as the transformation times became unduly long (*see* Fig. 7). A sample treated at 450° C transformed to bainite in 1200 s, but below this temperature the reaction time increased considerably. At 400° C 60% of the transformation occurred during the first 300 s, yet after 400,000 s no further transformation had occurred. This seemed to be the limit of transformation at this temperature and hence a sample for the extraction of carbides was transformed at 400° C for 300 s. Samples at 300° and 350° C were similarly treated until the reaction ceased, which was after 340 and 570 s respectively.

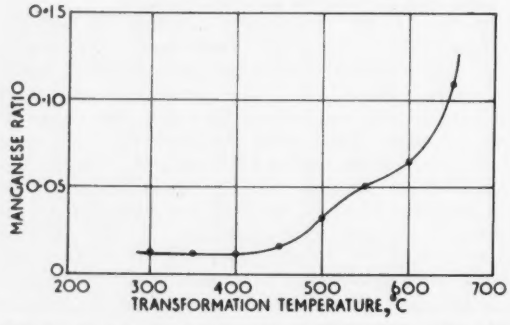

Fig. 4—Variation in the ratio Mn/Fe + Mn with isothermal transformation temperature for steel *MS*1 (0·33% C; 1·63% Mn)

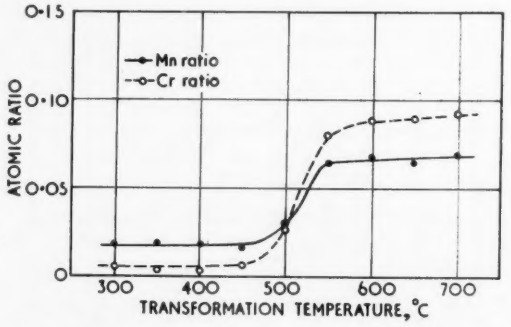

Fig. 6—Change in composition of the carbide phase with the variation in temperature of isothermal transformation for steel *CR*8 (0·36% C; 0·97% Cr; 0·78% Mn)

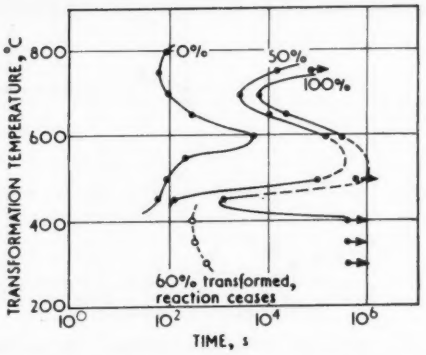

Fig. 7—Isothermal transformation diagram for steel *MO2* (0·45% C; 2·42% Mo)

The molybdenum content of the carbide is expressed as the ratio Mo/Fe + Mo (*see* Fig. 8). At all temperatures in the pearlite range (600–700° C), a carbide was formed which contained about 30% of molybdenum and had approximately the same lattice spacings as the carbide Mo_2C. Carbides from a sample transformed at 450° C gave a clear X-ray pattern corresponding to the carbide Fe_3C. The incompletely transformed samples produced in the temperature range 300–400° C also contained Fe_3C; some rather diffuse additional lines were attributed to the presence of Fe_2C.[12] The carbides formed in the bainite range contained only a small proportion of molybdenum.

3% Chromium Steels *CR4* and *CR5*

The transformation characteristics of steel *CR5* have been studied by a previous worker,[9] and the isothermal transformation curve which he obtained (Fig. 9) was determined for samples austenitized at 950° C for 4 h. No change was detected in the transformation characteristics of the material when the austenitizing treatment was ½ h at 950° C. The isothermal transformation curve for steel *CR4* is reproduced as Fig. 11. For these steels the chromium content of the carbide

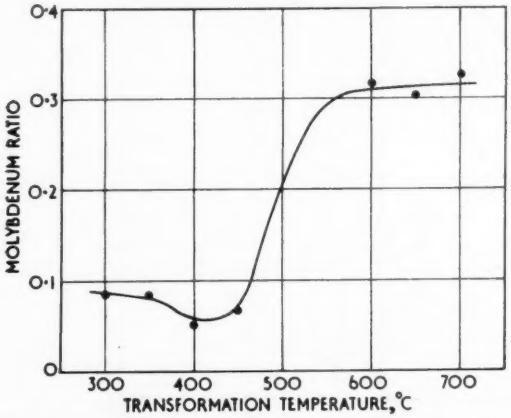

Fig. 8—Variation in the ratio Mo/Fe + Mo with isothermal transformation temperature for steel *MO2* (0·45% C; 2·42% Mo)

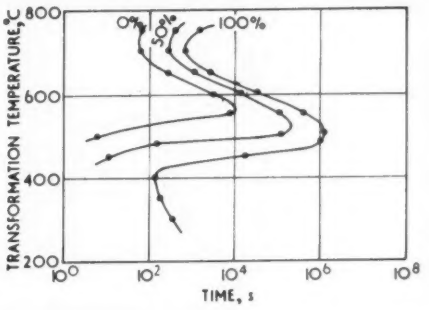

Fig. 9—Isothermal transformation diagram for steel *CR5* (0·22% C; 3·03% Cr)

is expressed as the ratio Cr/Fe + Cr, the manganese content being negligible.

For steel *CR4* the chromium content of the carbides obtained from samples completely transformed in the pearlite range was approximately constant. As the temperature was reduced from 500° to 450° C the transformation became more rapid, the product being bainite. This change in temperature also produced a reduction in the chromium content of the carbide and an abrupt increase in the hardness of the transformed specimen. The bainite residues contained only a small percentage of chromium but some concentration of chromium into the carbide phase was detected. Fe_3C is formed in the bainite range and Cr_7C_3 in the pearlite range.

The variation of carbide composition with temperature of isothermal transformation is very similar for steel *CR5*. Above the ' nose ' of the isothermal transformation curve, Cr_7C_3 of constant composition is produced and at lower temperatures cementite is formed. Both Fe_3C and Cr_7C_3 were identified in a sample transformed at 480° C.

The effect of tempering on the chromium content of the carbides is shown in Figs. 13 and 14. As tempering proceeds, some of the iron in the carbide is replaced by chromium and the orthorhombic structure persists until the carbide contains 19–20% of chromium. On further tempering, the Fe_3C gradually disappears and is replaced by Cr_7C_3. The rate of increase of chromium in the carbide is most rapid in

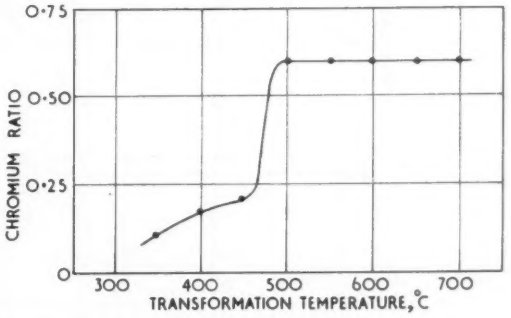

Fig. 10—Variation in chromium content of the carbide with temperature of isothermal transformation for steel *CR5* (0·22% C; 3·03% Cr)

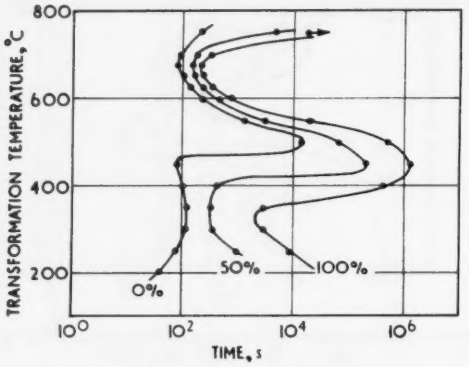

Fig. 11—Isothermal transformation diagram for steel *CR*4 (0·51% C; 2·97% Cr)

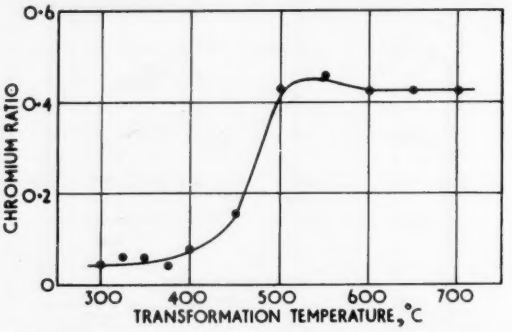

Fig. 12—Variation with temperature of isothermal transformation of the ratio Cr/Cr + Fe for the carbides extracted from steel *CR*4 (0·51% C; 2·97% Cr)

the early stages of tempering. The changes also proceed more rapidly at higher tempering temperatures. It is interesting to note that the maximum amount of iron present in the Cr_7C_3 type of carbide can be as high as 66%, and Fe_3C can contain up to 20% of chromium.

The values given in Fig. 14 for the chromium content of carbides extracted from samples of steel *CR*4 tempered at 400° C indicate that chromium diffuses into the carbide even at this low temperature. The process is extremely slow, however, and after 54 days the chromium ratio has only risen to 0·17.

The hardness values obtained from tempered samples of these chromium steels have been plotted (*see* Fig. 15) against $T(C + \log t)$ in the manner reported by Holloman and Jaffe[15] where

T = Absolute temperature, ° K
t = Time, s
C = Constant (5·55 for *CR*5 and 11·5 for *CR*4)

The hardness falls off as the carbide spheroidizes and takes up more chromium from the ferrite.

The carbide contains more chromium after prolonged tempering than after isothermally transforming to pearlite at the same temperature. Thus it appears that the carbide formed in the pearlite reaction is not in equilibrium. To find out what would happen if pearlitic samples were soaked for a long time at a high subcritical temperature, two samples, one from each of the steels *CR*4 and *CR*5, were isothermally transformed at 700° C and then soaked at 610° C for 30 days. A similar pair of samples was quenched and tempered at 610° C for 30 days. Carbides were extracted from these samples and analysed as in the previous experiments; the results obtained

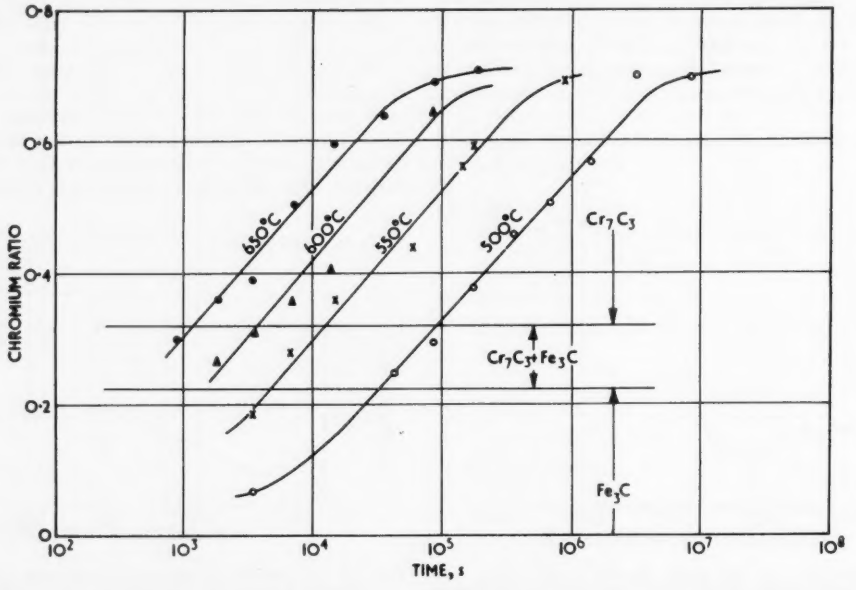

Fig. 13—Variation in chromium ratio with tempering time at various temperatures for steel *CR*5 (0·22% C; 3·03% Cr)

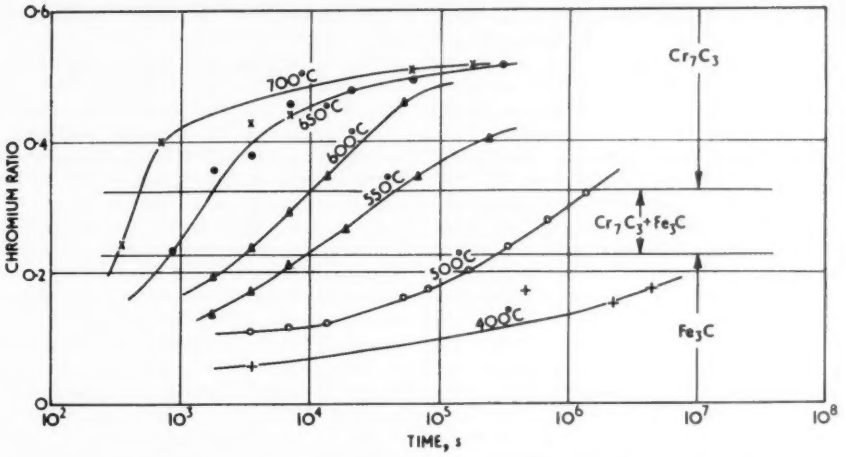

Fig. 14—Variation in chromium ratio with tempering time at various temperatures for steel *CR*4 (0·51% C; 2·97% Cr)

are recorded in Table II. The carbides extracted from the quenched and tempered samples have attained the high chromium content expected from previous experiments (*see* Figs. 13 and 14), whilst treating the isothermally transformed samples has only increased the chromium content of the carbide by a small amount. Although subcritical treatment of the isothermally transformed samples has produced a further diffusion of chromium into the carbide phase, equilibrium has not been reached. The isothermal transformation of these steels at 700° C produced a very coarse carbide structure and the distance over which the chromium was diffusing was greater than for the quenched steels; hence more chromium has diffused into the carbide phase in the quenched samples.

DISCUSSION OF RESULTS

The present work shows that iron carbide is formed in the bainite reaction. This is in agreement with previously published data. There is some indication however that the alloy content of the bainitic carbide is not necessarily the same as in the parent austenite. The concentration of alloying elements into the carbide is most marked in the upper bainite range, where long transformation times at the maximum temperature of bainite formation produces segregation of chromium and manganese into the carbide phase.

In those steels which contain sufficient alloying additions the carbides formed in the pearlite reaction are of approximately the same composition whatever the temperature of formation. Steel *CR*4 is a good example of this (*see* Fig. 11). It appears that in these steels the pearlite reaction is controlled by the rate of segregation of the alloying elements. Steels such as the 1½% manganese steel *MS*1 (*see* Fig. 4) transform more rapidly in the lower pearlite range and the amount of manganese in the carbide decreases as the transformation temperature is lowered. Steels with a high proportion of alloying element to carbon form an alloy carbide in the pearlite range.

During the tempering of a steel containing chromium, the alloying element segregates into the carbide phase. The carbide retains its orthorhombic structure up to a saturation value of 19–20% of

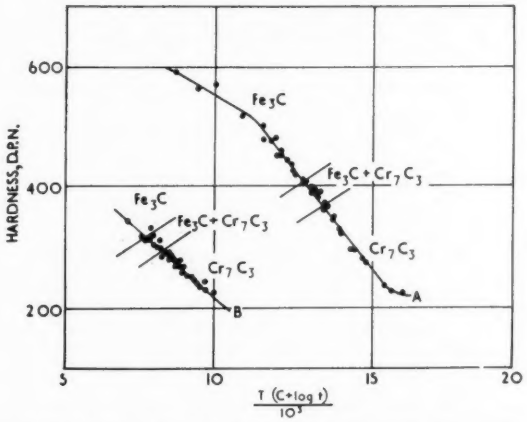

Fig. 15—Variation in hardness with degree of tempering (*A*) steel *CR*4, (*B*) steel *CR*5

Table II

CHROMIUM RATIO FOR SOME CARBIDES EXTRACTED FROM STEELS *CR*4 AND *CR*5

Heat-Treatment	Ratio of Chromium Atoms in Carbide Cr/Cr + Fe	
	*CR*4	*CR*5
Isothermally transformed at 700° C	0·43	0·61
Isothermally transformed at 700° C and subsequently tempered for 30 days at 610° C	0·44	0·63
Quenched and tempered at 610° C for 30 days	0·51	0·71

chromium. On further tempering some of the Fe_3C disappears and is replaced by Cr_7C_3. As the chromium content of the carbide increases, more Cr_7C_3 forms and the amount of Fe_3C decreases until only the trigonal carbide remains; the chromium content of the Cr_7C_3 increases during subsequent tempering until the equilibrium composition is attained. It appears that the trigonal carbide can contain as little as 28 wt-% Cr, slightly less than previously reported.[1]

Balluffi, Cohen, and Averbach[14] suggest that, since the Fe_3C forms as a large number of plate-like particles and the Cr_7C_3 appears as a smaller number of spherical particles, the nucleation of Cr_7C_3 must be independent of the Fe_3C particles already present. This evidence is rather inconclusive since, in order to reduce the total surface energy of the system, the carbide will spheroidize if tempered for a sufficiently long time, even if no change in crystal form occurs; the large particles will tend to grow and spheroidize, while the smaller particles dissolve in the ferrite matrix.

Alternatively, it is possible that, when the Fe_3C lattice becomes saturated with chromium, it becomes unstable and breaks down to form the trigonal carbide and reject iron atoms. If the reaction is

$$\underset{\text{(orthorhombic)}}{Fe_7Cr_2C_3} \rightarrow 2Fe + \underset{\text{(trigonal)}}{Fe_5Cr_2C_3}$$

the orthorhombic carbide would become unstable when it contained 19·7% of chromium, and the trigonal carbide formed would contain 24·8% of chromium. This agrees quite well with the composition changes established by experiment if some allowance is made for the diffusion of chromium into the carbide during the reaction. Since the transformation will start at the surface of the carbide particle where the chromium concentration is highest, the rejection of iron atoms will be through a surface layer of trigonal carbide. No metallographic evidence has been found to support this theory.

The essential difference between these two theories is the mechanism of nucleation of Cr_7C_3. One theory proposes that a nucleus for the alloy carbide is formed when metal and carbon atoms come together to form a particle of the trigonal carbide of a certain minimum size, and it seems probable that the ideal position for the formation of such a nucleus will be the site occupied by an iron carbide particle which has become to small to remain stable. The composition of Cr_7C_3 nucleated on such a site would tend towards that observed in practice and, due to the local segregation of chromium and carbon, the probability of nucleation will be high. The other theory suggests that the Cr_7C_3 structure is more easily generated at the surface of the Fe_3C already present by reorientation of the orthorhombic lattice and rejection of iron atoms. With the information at present available it is not possible to decide whether both processes contribute to the nucleation of the trigonal carbide or whether one of these mechanisms predominates.

CONCLUSIONS

(1) There is some concentration of the carbide-forming elements chromium, molybdenum, and manganese into the carbide phase during the bainite transformation, especially at temperatures in the upper bainite range. The steels investigated formed only iron carbides in the bainite transformation.

(2) In the very low-alloy steels investigated, the alloy content of the iron carbide formed in the pearlite increased progressively with increasing transformation temperature. Steels of higher alloy content transformed very slowly in the lower pearlite range and the alloy content of the pearlitic carbide was approximately the same at all temperatures. Alloy carbides were formed in those steels which had a sufficiently high ratio of alloying element to carbon.

(3) In the tempering of chromium steels the rate of increase of chromium in the carbide decreases as tempering proceeds and the reaction rate increases as the temperature is raised. Some diffusion of chromium into the carbide is detectable even at tempering temperatures as low as 400° C. If sufficient chromium is present the trigonal carbide begins to form when the Fe_3C contains 19–20% of chromium; the trigonal carbide can contain as little as 28% Cr. The mechanism of the change from Fe_3C to Cr_7C_3 is uncertain.

(4) In the 3% chromium steels studied, the chromium content of the carbides formed in the pearlite transformation was less than the value obtained on tempering at the same temperature. It appeared that the size and distribution of the carbide particles were important factors in determining the rate at which chromium diffused into the carbide.

Acknowledgments

The author wishes to express his gratitude to Professor A. G. Quarrell and Emeritus Professor J. H. Andrew for their guidance and encouragement at various stages of this work. Thanks are also due to the author's colleagues in the University of Sheffield for their valuable support and suggestions and to the British Iron and Steel Research Association whose financial support made this work possible.

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The System $CaO–MgO–Al_2O_3–SiO_2$

PHASE-EQUILIBRIUM RELATIONSHIPS ALONG THE JOIN ANORTHITE–SPINEL

By J. H. Welch, M.A.

SYNOPSIS

The paper describes the results of experimental work on the join anorthite–spinel within the quaternary system $CaO–MgO–Al_2O_3–SiO_2$. It is shown that anorthite and spinel do not form a binary system because corundum occurs as a primary phase, and that this fact contradicts recently published data on the extent of the primary phase field for mullite within the system $CaO–Al_2O_3–SiO_2$. The paper also describes a number of variations in the normal experimental technique, which increased the speed and facility of the investigation; these included the use of an improved method of thermal analysis, the use of a heated-stage microscope for exploratory work, and the adoption of a 5% Rh–Pt/20% Rh–Pt thermocouple for temperature measurement. 1162

Introduction

THIS INVESTIGATION has been pursued with the principal aim of securing further information about the location of the primary phase volumes within the quaternary system $CaO–MgO–Al_2O_3–SiO_2$. Knowledge of the four faces of the tetrahedron is fairly complete, although certain modifications have been, and may still be, proposed in the light of more recent investigation. Within the tetrahedron itself information on the demarcation of the phase volumes is more limited, as the gathering of data required for this type of equilibrium study is a lengthy and laborious process. For this reason investigators have confined their studies to such portions of the system as will repay the least effort with the maximum information in the region in which their particular interest lies.

Little is known of the region towards the Al_2O_3 apex of the tetrahedron and it was decided to explore the plane $CAS_2–C_2AS–MA$* for which preliminary knowledge of the two-component systems $CAS_2–C_2AS$, $C_2AS–MA$, and $CAS_2–MA$ was required (*see* Fig. 1). The first system was studied by Rankin and Wright,[1] and the second, more recently, by Nurse and Stutterheim[2] and, independently, by Prince.[3] The third system is the subject of the present investigation, and besides having an affinity with the above work, the results should be of interest in studies on certain blast-furnace slags and refractory materials. Moreover, although the possibility existed that the join $CAS_2–MA$ might be of a simple binary nature as is the case with $C_2AS–CAS_2$ and $C_2AS–MA$, there appeared to be a strong likelihood that intrusion of other primary phase fields might occur. According to the data of Rankin and Wright on the base plane $CaO–SiO_2–Al_2O_3$ the occurrence of corundum would not be unexpected; other possibilities occur as a result of more recent work by Filonenko and Lavrov,[4] establishing a primary phase field for CA_6, and by the extension of mullite into the formerly established field of corundum, according to Toropov and Galakhov.[5] Any evidence obtained during the present work, corroborating these later findings, would therefore be of especial significance.

Throughout this work a secondary aim has been the practical testing of a number of variations on normal experimental technique, intended to increase the speed and facility of the investigation, and these are described in the following section.

EXPERIMENTAL PROCEDURE

Liquidus temperatures towards the spinel end of the $CAS_2–MA$ join are far outside the range of the quench furnaces used, so that studies were limited to compositions between 100% CAS_2 and 40% CAS_2–60% MA. Fourteen mixtures in this range were prepared from reagents whose analyses are given in Table I. The correct proportions of these materials were weighed and then mixed in a mechanical shaker for several hours. The mixtures, which were designed to yield 10 g of melt, were fired at 1600° C in platinum vessels in an electric furnace with a rhodium–platinum resistance element. After cooling, the melts were broken up and reduced in a steel percussion mortar to pass the 100-mesh sieve. One 6-h firing was usually sufficient to achieve complete reaction and homogeneity of the glass, as determined by the uniformity of refractive index, but the process was repeated where necessary. No other form of grinding was used since contamination from agate, corundum, or tungsten carbide mortars is irremovable. A very efficient, mechanically operated, magnetic separator was used to remove steel particles from the preparations. The preparations were passed twice through the separator and, as a final precaution, were again mixed for some hours on the mechanical shaker. This was done to obtain a uniform distribution of the material should any slight lack of homogeneity have escaped detection. The effectiveness of this treatment in promoting uniformity was confirmed by the subsequent investigation.

* The usual abbreviations of the formulæ of silicate compounds are used here: anorthite, $CaO.Al_2O_3.2SiO_2 = CAS_2$; spinel, $MgO.Al_2O_3 = MA$. The pure oxides, however, are always written in full: corundum = Al_2O_3.

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Table I

ANALYSES OF STARTING MATERIALS

n.d. = Not determined

Material	CaO	MgO	SiO_2	Al_2O_3	(NaK)O	FeO	Fe_2O_3	TiO_2	SO_3	Loss at 1200° C	Purity*
$CaCO_3$	56·16	0·01	n.d.	0·03	0·09	n.d.	0·01	Nil	0·01	44·06†	99·75
MgO	0·07	n.d.	0·25	0·04	n.d.	n.d.	0·046	<0·01	0·34	7·74	99·55
SiO_2	n.d.	n.d.	99·95	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	Nil	99·95
Al_2O_3	0·044	n.d.	0·02	n.d.	0·234	<0·001	0·026	Nil	0·079	0·34	99·68

* Determined by difference (except SiO_2) † Nil at 110° C

From a preliminary examination the most promising field for study appeared to be in the range 100% CAS_2 to 80% CAS_2–20% MA, and the initial mixtures were prepared accordingly. Rapid exploratory work on these mixtures led to the preparation of further suitable mixtures, and eventually a representative range of mixtures was prepared for detailed study by the quenching method.

The exploratory work was carried out with a heated-stage microscope, which has been fully described elsewhere,[6] and also by means of heating curves. The method of thermal analysis used involved continuous automatic recording of specimen temperature and difference temperature by a conventional photographic recorder. The mounting of the specimen, however, was unorthodox and a brief description may be of interest. A normal assembly for obtaining heating curves is illustrated in Fig. 2; two platinum crucibles are used containing, respectively, the sample and an inert reference material, usually corundum. The difference temperature is obtained from thermocouples, connected in series opposition, immersed one in each of the crucibles. The absolute temperature of the sample is obtained from a separate thermocouple in the sample crucible, and the absolute temperature and difference temperature may be recorded by separate reflecting galvanometers in a photographic recorder. It is possible to use only one thermocouple in the sample crucible to perform the dual function of absolute and difference temperature measurement. In this case, if deflection instruments are used, current flowing in the absolute-temperature circuit will affect the measurements obtained from the difference circuit and vice versa. To prevent interaction occurring in this way, discontinuous recording of the two circuits may be used; alternatively, null-balancing measuring instruments which draw no current from the thermocouples may be substituted.

A disadvantage of the normal method is that about 2 g of melt is usually required in the sample crucible and its subsequent extrication is a matter of some difficulty. This applies also to the thermocouples unless a protective sheath, which results in greatly reduced sensitivity, is used. The sacrifice of a short length of the thermocouples can be tolerated if narrow-gauge wire is used, but the consequent lack of rigidity produces another difficulty: surface tension effects within the melt can be very powerful and may cause complete displacement of the thermocouples from the melt. Sheathing the thermocouples may mitigate this trouble also, but the normal heating-curve crucible assembly still could not be used with certain melts in which, in the author's experience, surface-tension effects have caused the melts to flow upwards for a considerable distance and out of the crucible altogether. It was to overcome these difficulties, and the progressive damage to crucible, sheath, or thermocouple incurred through the use of the normal method, that an alternative arrangement was sought.

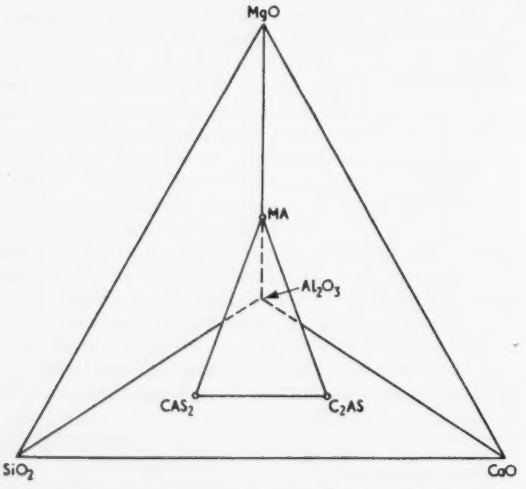

Fig. 1—Relation of system anorthite–spinel (CAS_2–MA) to quaternary CaO–MgO–Al_2O_3–SiO_2

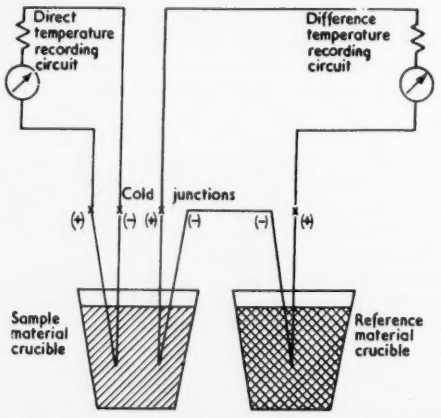

Fig. 2—Normal arrangement for thermal analysis

Unpublished work by Prodan, referred to by Bogue,[7] describes a method of obtaining heating curves using small amounts of material attached directly to the junction of a thermocouple. Even after complete or partial melting the sample is retained on the thermojunction by surface tension. Prodan used sample weights varying between 0·1 and 0·5 g and thermocouple wire of 0·8 mm dia. The melts studied by Prodan contained iron and caused rapid deterioration of the thermojunction, and so sample temperatures had to be measured indirectly by a process of calibration using a separate thermocouple situated near to but not in contact with the melt. The assembly occupied very little space in the furnace and multiple thermal analysis could be attempted simultaneously on as many as six samples.

The method adopted in the present work was basically similar to Prodan's. The most important modification was the device used to obtain continuous recording of sample temperature and difference temperature, which avoids certain difficulties or complications associated with the normal method as described earlier. The operation of this device is shown in Fig. 3. Although a single thermocouple in the sample measures both absolute and difference temperatures, the measuring circuits are isolated from each other by a synchronous vibrator. The rapid switching of the vibrator effectively produces continuous recording, and avoids any possibility of mutual interaction through the use of deflection instruments with a common thermocouple. The vibrator consists of a non-polarized relay operating at twice the frequency of the mains supply in order to minimize the chance of stray alternating currents being picked up and rectified in the measuring circuits. Capacitors are used in the measuring circuits to absorb fluctuations due to the use of the vibrator; although the capacitors

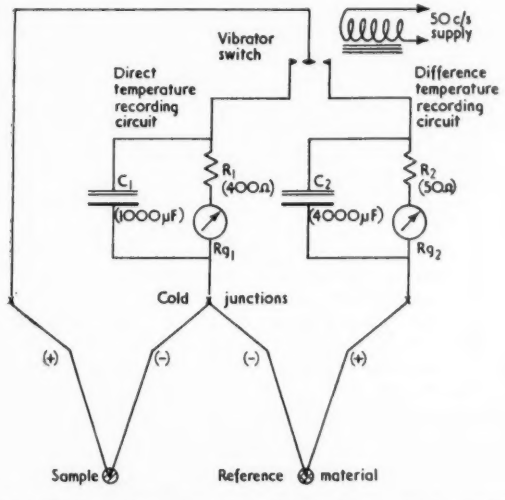

Fig. 3—Arrangement for thermal analysis with materials attached directly to thermocouple. Values for the circuit components used are not critical, provided that $(R + Rg) \times C \geqslant 200{,}000$, R and C being expressed in ohms and microfarads, respectively

are electrolytic, no spurious voltages attributable to their use have been detected, a fact which may possibly be explained by the fairly low resistance of the measuring circuits.

The thermocouple wire used has a diameter of 0·2 mm. The sample material is moistened with water and applied to the thermojunction with a spatula, and the reference junction is similarly coated with a bead of alumina cement. The beads are gently warmed and, when they are almost dry, can safely be transported to the furnace. The average weight and diameter of a sample bead are 0·05 g and 3·0 mm, and consequently its heat capacity is very small. The heating rate of the furnace must therefore be unusually free from fluctuations which might be reflected in the recording circuits. To achieve this, and also to prevent pick-up of stray furnace currents, the beads are enclosed in a platinum crucible with lid and the thermocouple insulators are sheathed in platinum foil. This electrical shielding is then connected to a suitable point, not necessarily at earth potential, which is best determined by experiment. In the present instance connection to the negative side of the furnace controller input circuit was found to reduce electrical leakage in the thermocouple circuit to a minimum.

The very small size of the sample leads to a reduction in sensitivity which, however, is not so marked where comparison is made with a normal crucible assembly employing sheathing for the thermocouples. On the other hand, the small sample size and intimate thermocouple contact are a definite advantage in rapidly establishing thermal and chemical equilibrium and in countering the problem of drift. The removal of the specimen is accomplished by twisting the thermocouple above the bead and cutting through the twisted portion; the thermojunction is then re-welded and pulled down to its former level ready for the attachment of a new specimen. The thermocouple can be calibrated *in situ* by temporarily introducing a reference thermocouple. Any sudden or progressive change in the calibration can be observed and estimated from the change in the static e.m.f. of the difference circuit.

The quench furnaces used in this work had a 20% Rh–Pt internal winding which was suitably spaced so as to produce a hot zone 5 cm long, uniform to $\pm 1°$ C. Their maximum working temperature is believed to be about 1800° C but their normal operating limit has been confined to 1775° C. The furnace controllers, which have been described elsewhere,[8] permitted short-term temperature control to $\pm 0{\cdot}1°$C, but manual adjustment was required to compensate for drift caused by changes in the furnace winding during lengthy heat-treatments. This well-known feature of furnace drift was put to practical use under certain experimental conditions. In others it could be arrested or even reversed as desired by the inclusion of an adjustable programme control. A double quenching rig enabled two charges to be suspended in the furnace and separately quenched; by arranging for two charges to share a common heat treatment wherever possible, appreciable time was saved.

The International Temperature Scale of 1927, for which the defined values of the melting points of gold,

Table II

THERMAL DATA

All temperatures are °C (International Temperature Scale 1927)

Composition, wt.-%		Limiting Quench Runs			Phases Observed*				Relevant Boundary in Fig. 3	Location of Boundary by:		
CAS_2	MA	Starting Temperature	Duration of Run, h	Quenching Temperature	CAS_2	Al_2O_3	MA or MA–Al_2O_3 Solid Solution	Glass		Quenching	Heated-stage Microscope	Thermal Analysis
100	0	...	...	...	...	...	...	...	AC	...	1550	1550
97·5	2·5	1539	½	1539	P	...	...	P	AC	1540	1542	1540
		1539	½+½	1541	...	...	...	P				
		1505	16	1518	P	P	...	P	BC	1520	...	...
		1505	16+6	1522	P	...	...	P				
		...	...	...	...	...	...	...	DG	...	...	1467
95	5	1530	5	1530	...	...	...	P	AC	1528	1525	1529
		1530	5+2	1525	P	...	...	P				
		1515	1½	1515	P	P	...	P	BC	1520	...	...
		1525	16	1520	P	T	...	P				
		...	...	...	...	...	...	...	DG	...	...	1471
90	10	1530	25	1530	...	P	...	P	CH	1532	...	...
		1530	25+16	1535	...	...	...	P				
		1505	2	1505	P	P	...	P	CE	1508	1514	1508
		1510	18	1510	...	P	...	P				
		1479	41	1455	P	P	...	P				
		1479	46	1450	P	(P)	P	(P)	DG	1452	...	1462
		1451	1	1451	P	...	P	...				
		1451	1+1	1475	P	(P)	P	(P)				
85	15	1560	20	1550	...	...	...	P	CH	1550	...	...
		1545	4	1545	...	P	...	P				
		1545	4+2½	1550	...	T	...	P				
		1472	1	1472	...	P	...	P	CE	1471	1473	...
		1472	1+½	1470	P	P	...	P				
		1460	1+½	1453	P	P	...	P	DG	1452	1452	1460
		1449	2+½	1451	P	(T)	P	(T)				
83	17	1552	5	1552	...	P	...	P	CH	1554	...	...
		1552	5+2½	1556	...	...	...	P				
		1470	½	1470	...	P	...	P	EH	1467	...	...
		1470	½+1	1465	...	P	P	P				
		1459	1+1	1455	...	P	P	P	DG	1453	...	1455
		1451	1	1451	P	(T)	P	(T)				
80	20	1560	2	1560	...	...	...	P	CH	1558	...	...
		1574	21	1555	...	P	...	P				
		1530	16	1523	...	P	...	P	EH	1522	...	...
		1530	16+1	1522	...	P	T	P				
		1479	41	1455	...	P	P	P	DG	1452	...	1460
		1479	46	1450	P	(P)	P	(P)				
75	25	1584	1	1584	...	...	...	P	HJ	1582	1584	...
		1584	1+1	1580	...	...	P	P				
		1540	3	1541	...	...	P	P	HF	1540	...	...
		1540	3	1540	...	T	P	P				
		...	...	...	...	...	...	...	DG	...	...	1464
72·5	27·5	1610	½	1610	...	...	...	P	HJ	1608	1608	...
		1610	½+2	1606	...	...	P	P				
		1515	2	1515	...	P	P	P	HF	1520	...	...
		1520	3	1520	...	T	P	P				
		1520	3+4	1525	...	...	P	P				
70	30	1643	½	1643	...	...	...	P	HJ	1640	1643	...
		1638	½	1638	...	...	P	P				
		1510	16	1505	...	...	P	P	HF	1500	...	...
		1500	2	1500	...	T	P	P				
		1480	1+2	1495	...	P	P	P				
		1479	41	1455	...	P	P	P	DG	1452	...	1460
		1479	46	1450	P	(P)	P	(P)				
65	35	...	...	...	...	...	...	...	HJ	...	1697	...
		1473	1	1473	...	P	P	P	HF	1474	...	...
		1473	1+2	1476	...	...	P	P				
		...	...	...	...	...	...	...	DG	...	...	1458
60	40	...	...	...	...	...	...	...	HJ	...	1740	...
		1470	16	1463	...	...	P	P	HF	1458	...	...
		1470	16+2	1458	...	T	P	P				
		1479	41	1455	...	(...)	P	P	DG	1452	...	1463
		1479	46	1450	P	...	P	(P)				
50	50	...	...	...	...	...	...	...	HJ	...	>1800	...
		1454	½	1454	...	...	P	P	DG	1452	...	1455
		1454	½+½	1450	P	...	P	...				
40	60	1454	½	1454	...	...	P	P	DG	1452	...	1455
		1450	½	1450	P	...	P	...				

* P = present, T = trace only. The use of brackets signifies that the presence or absence of a phase is assumed to be the result of disequilibrium

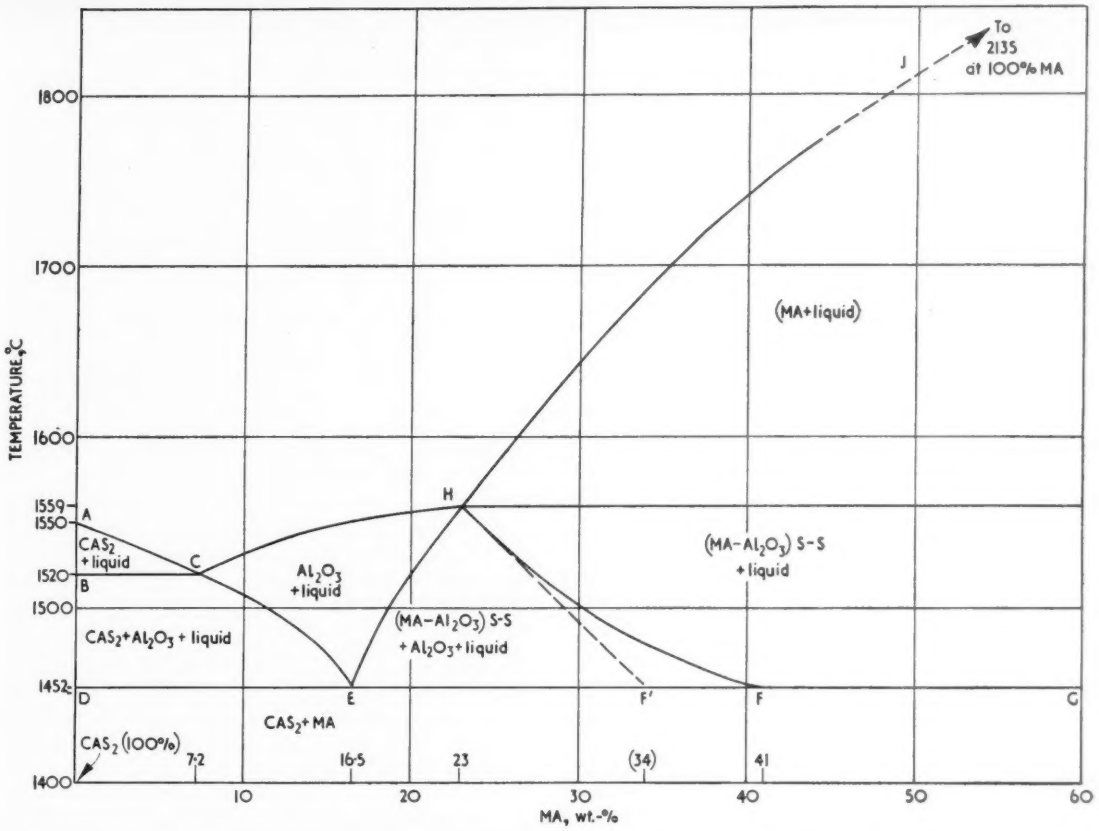

Fig. 4—Temperature/composition diagram for the join anorthite–spinel giving qualitative indication of phases present

palladium, and platinum are 1063°, 1555°, and 1773° C, was used throughout the present work. Temperature measurements were made with a 5% Rh–Pt/20% Rh–Pt thermocouple which had been calibrated at 20° intervals from a normal Pt/10% Rh–Pt thermocouple, and also at the three fixed points mentioned above.

One of the advantages of this particular thermocouple arises from the shape of the e.m.f./temperature curve. At temperatures higher than 1400° C the curve becomes linear with a constant slope of about 10μV/° C. Consequently the determination of the palladium and platinum points enables extrapolated temperatures to be obtained with far greater accuracy than when using a thermocouple having an approximately parabolic e.m.f./temperature relationship. At room temperatures, however, the output of the thermocouple is very small indeed. This means that a large variation in cold-junction temperature will have very little effect on the accuracy of high-temperature measurements. As an example, ignoring a cold-junction temperature of 20°, 30°, and 50° C would affect the accuracy of any temperature measurement above 1300° C by only ½°, 1°, and 2° C, respectively.

The principal advantage of this combination, however, is its greatly improved stability with time of use, and its adoption was originally inspired by the recent trend towards the avoidance of pure Pt/Rh–Pt thermocouples.[9] Only one very brief, and frequently misquoted, reference to its previous use has been found in the literature.[10] Records kept during the present work show that 1250 h of use at temperatures in the range 1300–1700° C produced only a 5° change in calibration. The couple was rechecked after every 50 or 100 h and never showed an alteration amounting to more than 1° C for every 100 h of duty. There seemed to be an established trend for the e.m.f. to decrease with time at temperatures below 1500° C and to increase at temperatures above 1700° C. A comparative run at 1600° C and of 100 h duration was made between a normal Pt/10% Rh–Pt couple and a 5% Rh–Pt/20% Rh–Pt couple; whereas the calibration of the former had altered by 11° C, no detectable change had occurred in the latter. Unfortunately the wire batches from which the first set of 5% Rh–Pt/20% Rh–Pt couples was made were several years old and the rhodium then used in their manufacture contained up to 0·1% iron as an impurity. This fact was discovered when thermocouples, made from recently prepared batches of wire of high purity, showed a considerable departure from the previous calibration curve. It remains to be seen

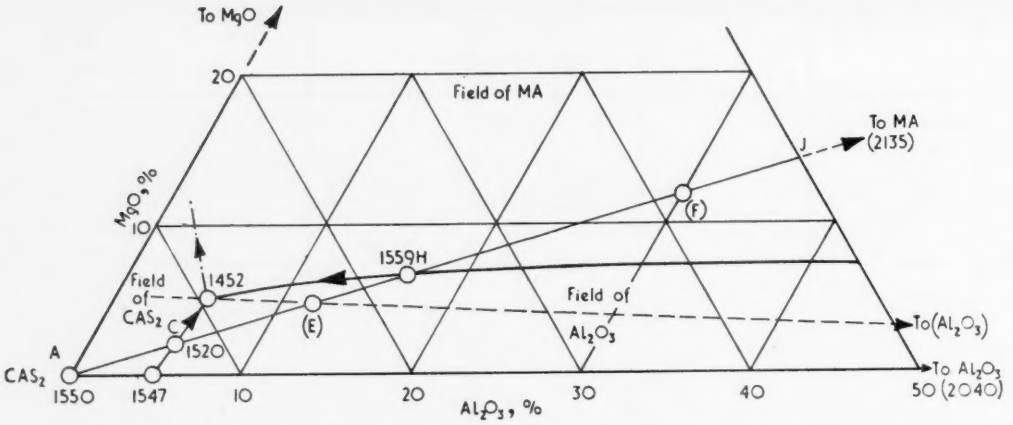

Fig. 5—A portion of the plane CAS_2–Al_2O_3–MgO showing a part of the join CAS_2–MA and probable location of four-phase boundary CAS_2, MA, Al_2O_3, and liquid

to what extent the previously discovered properties of this type of thermocouple will be modified by the use of purer materials, but it seems likely that still further improvement may be expected rather than the reverse.

DISCUSSION OF RESULTS

The results obtained from quenching, thermal analysis, and the heated-stage microscope observations are presented in Table II. It is not possible to record there the complete thermal history for individual quenching runs. Only limiting quenching runs are recorded and, when the starting and quenching temperatures are the same, a single figure in the 'Duration of Run' column indicates the number of hours run at constant temperature. When they are different, a single figure indicates a gradual transition, usually over a long period, to the quenching temperature. Two figures indicate a preliminary heat-treatment at the starting temperature, followed by a further run at the quenching temperature. It will be seen that no distinction is made between MA or MA–Al_2O_3 solid solution as separate phases, and optically the recognition of them as such would in most cases be impossible. It is assumed that a varying amount of Al_2O_3 is present in solid solution with MA in the temperature range 1452–1559° C; pure MA only occurs when accompanied solely by CAS_2 at temperatures below 1452° C, or in contact with liquid at temperatures above 1559° C.

The phase relationships are depicted in Fig. 4, but this diagram cannot of course be used to express quantitatively the phases present in the system since this is non-binary. Because of the intrusion of corundum and the occurrence of spinel–corundum solid solution, the system can best be understood as a part of the join plane CAS_2–MA–Al_2O_3, within the plane CAS_2–Al_2O_3–MgO, as shown in Fig. 5. For compositions on the line JH, pure spinel crystallizes as the primary phase until the temperature falls to 1559° C, the compositions of the liquid travelling along the line JH and arriving at H at this temperature. As the temperature continues to fall corundum is co-precipitated with the spinel as a solid solution. The corresponding liquid compositions follow the corundum–spinel boundary, but corundum is not precipitated as a separate phase until saturation of the solid solution is reached; this takes place at a particular temperature for each starting composition which is given by the curve HF in Fig. 4. Roy *et al.*[11] have described the system Al_2O_3–MA, and their data for the solid-solution saturation curve provide a check on the accuracy of the boundary HF in Fig. 4. The course of the boundary deduced from their work is shown as the dotted line HF' and it will be seen that close agreement is obtained at temperatures between 1559° and 1500° C. The increasing discrepancy at lower temperatures may in the greater part be attributed to lack of equilibrium obtained in the present work. This would be caused by the failure of spinel, when present in large amounts, to participate fully in the solid-phase reaction to produce complete saturation. On the other hand, Roy's technique, of fusing and quenching specimens and then annealing at lower temperatures until ex-solution of corundum occurred, might produce a degree of supersaturation which could contribute to the discrepancy. In any case, the latitude possible in drawing the ex-solution curve in the binary system Al_2O_3–MA from Roy's data, coupled with the degree of accuracy obtainable in the geometrical constructions involved, do not permit exact comparisons from a quantitative aspect of the data for the two systems. Nevertheless Roy's work gives a valuable qualitative check on the results obtained in the present investigation.

In Fig. 5 it is possible, by extrapolation, to deduce with reasonable accuracy the intersection of the four-phase boundary CAS_2–Al_2O_3–MA–liquid, which is a piercing point in the plane depicted, on the assumption that the MA–Al_2O_3 boundary follows the path shown. The composition of this piercing point was estimated to be 89·5% CAS_2–5·5% Al_2O_3–5% MgO. An experimental mixture of this composition did not melt completely to liquid at 1452° C. Quenching experiments showed that a trace of spinel–corundum solid solution remained and that the final melting temperature was

1462° C. The temperature of the spinel liquidus surface rises so steeply in this region, however, that it may be assumed that the composition quoted for the piercing point is not in error by more than 0·25% for any of the constituents. The form of the boundary curve Al_2O_3–*MA* assumed in drawing Fig. 5 could justifiably be inferred from the currently accepted representations of the major ternary systems bounding the quaternary. The consequent depiction of the corundum primary-phase volume as a thin, wedge-shaped layer on the CaO–SiO_2–Al_2O_3 face of the quaternary would generally be considered valid. However, Toropov and Galakhov[5] recently revised the system Al_2O_3–SiO_2 and discovered that mullite melts congruently, their observations being confirmed by Budnikov *et al.*[12] Furthermore, they stated that in the major ternary systems mullite occupies much of the field formerly attributed to corundum. The corner of the CaO–SiO_2–Al_2O_3 diagram with revisions by Toropov and Galakhov is reproduced in Fig. 6. In this plane the join CAS_2–Al_2O_3, which is also a projection on to the plane of the join CAS_2–*MA*, is no longer an Alkemade line. On this basis, therefore, the appearance of mullite was a reasonable expectation during the present work, but was never encountered. The intrusion of corundum into the CAS_2–*MA* join, which appears plausible enough in the original diagram, is quite inconsistent with the revised version, and confirms the existence of a common boundary between CAS_2 and Al_2O_3.

The appearance of CA_6 was anticipated as a further possibility which might occur during the investigation.

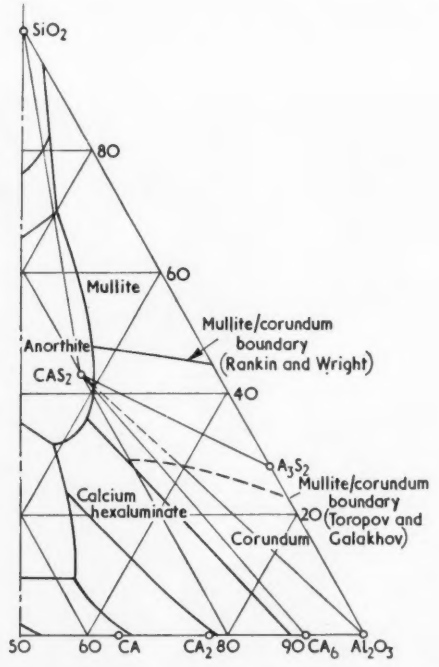

Fig. 6—A portion of the system CaO–SiO_2–Al_2O_3 based on current amendments to the original data of Rankin and Wright

The form in which corundum appeared in the quenched preparations consisted of thin plates, often of hexagonal outline, prominently displaying stepped triangular growth-planes (Fig. 7). In basal section these exhibited very low birefringence; nearly all the crystals polarized in greys but higher-order colours were exhibited by a few plates viewed on edge. From the optical data published by Filonenko and Lavrov[4] there seemed to be a very great risk of confusing the appearance of corundum and CA_6. A sintered preparation of the latter composition heated for two weeks at 1600° C yielded an X-ray diffraction pattern markedly different from that of pure corundum. The hexagonal plates occurring in the quenched glasses, however, were hand-sorted under the microscope, and the concentrated product gave a perfect corundum pattern.

The appearance of the spinel or, more accurately, corundum–spinel solid solution in the quenches was typical. When present as the primary phase the crystals were large well-formed octahedra. At temperatures in the vicinity of the solidus the crystals were aggregated in clusters of very small individuals. When interspersed with the smallest corundum crystals, the very low birefringence of the latter, and high refractive index of both, made it very difficult to distinguish them under the microscope.

In the quenched preparations anorthite was encountered only in the triclinic form. It appeared characteristically as sheets of multiple-twinned crystals, exhibiting under crossed nicols a striated appearance and inclined extinction (Fig. 8). Single crystals of the triclinic form grown on the heated-stage microscope consisted of perfect plate-like rhombs. Both the hexagonal and orthorhombic forms described by Davis and Tuttle[13] were produced from pure anorthite glass and the effects of different heat-treatment on the production of these polymorphs have been extensively studied with the aid of heating curves and the heated-stage microscope. The latter method has been of particular assistance in this work, of which a separate account will be published later; the scope and complexity of the sub-solidus reactions of anorthite precludes their discussion here.

The system CAS_2–*MA* is not easily susceptible to investigation by the conventional quenching technique. It is customary to quench melts from successively lower temperatures to locate first the liquidus and then the appearance of each successive phase until the solidus is reached. This is not always possible when a phase shows some reluctance to crystallize even when the melt is highly supersaturated. Anorthite can definitely be included in this category, at least as far as compositions covered in the present investigation are concerned. The alternative is to begin with fully crystalline material and quench from successively higher temperatures, noting the disappearance of each phase until the liquidus is reached. Corundum and spinel do not lend themselves to this treatment as their rate of dissolution is so slow that they may persist for long periods even when superheated. This difficulty does not arise when determining the liquidus temperature with the heated-stage microscope; the technique then consists of superheating the specimen considerably until only one very

small crystal is retained in the melt. The temperature is then lowered and adjusted until the crystal is seen neither to grow nor to melt, and in fact to be in equilibrium.

In dealing with the special difficulties of the system CAS_2–MA the following quenching technique was adopted. The chosen compositions were completely melted and quenched. The glass was then inoculated with one or two seed crystals of the appropriate phases, in some cases different seeds being used in separate packets on the double quenching rig. The rig was then suspended in the furnace at the required temperature. Even this treatment was not infallible since the spinel usually recrystallized to some extent as soon as the quench packet entered the furnace. The recrystallization product was so finely divided, however, that re-solution took place within a short time. Very long heat-treatments in the quench furnace were still necessary to obtain true equilibrium. Even so, many of the compositions produced disequilibrium in the region of the solidus temperature of the system. It was possible to obtain quenches containing anorthite, spinel, corundum, and glass, because of the failure of the corundum to react with the liquid on reaching the solidus. This difficulty did not occur with the compositions immediately adjacent to the CAS_2–MA–Al_2O_3 reaction-point composition, nor did it arise with compositions of over 40% MA content in which the corundum was all in solid solution at temperatures above the solidus. The solidus temperature determined from these compositions was 1452 $\pm$ 1° C, and the reaction-point composition ($CAS_2 + MA = Al_2O_3 +$ liquid) is estimated to be 83·5% CAS_2–16·5% MA ($\pm$ 0·5%).

For other compositions very slow cooling towards the solidus yielded equilibrium products only at 1455° C and additional disequilibrium phases at 1450° C. Fully crystallized compositions below the solidus remained unaltered by quenching from 1450° C, but traces of glass were apparent at 1455° C. The limiting approach to the solidus made from both directions with these compositions gives results in good agreement with the determinations made on the few compositions which behaved ideally. The latter gave concordant results in the determinations of the solidus by thermal analysis but, as might be expected, considerable variation occurred with the remaining compositions.

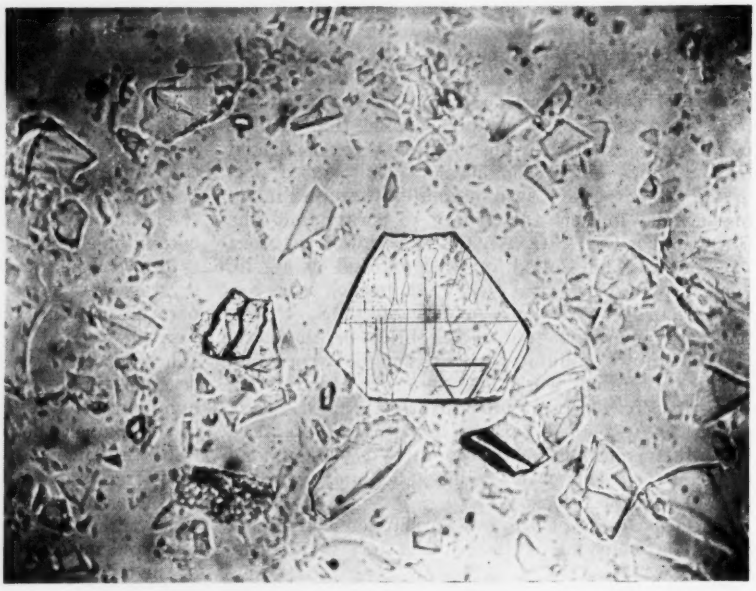

Fig. 7—Photomicrograph of crystalline corundum and glass. Normal transmitted light × 70

Fig. 8—Photomicrograph of triclinic anorthite. Plane polarized light, crossed polars × 70

The final melting of corundum and spinel was far too sluggish to be determined by thermal analysis, but the melting of anorthite was clearly marked. Solidus melting was measured from the peak of the break on the heating curve; intermediate and final melting points were measured from the termination of the corresponding break. This procedure is justifiable theoretically and in the present instance produces excellent agreement with the quenching results. The more common practice of selecting the peak of a break—where it can be distinguished at all—as representing a final melting point is unreliable. The conditions where a distinct peak occurs usually involve a melt that is being heated at a rate faster than the rate at which the dissolving phase can maintain equilibrium with its surroundings. It appears to be largely fortuitous that the peak of such a break may, and often does, give a good agreement with quenching determinations, since the degree of coincidence must be affected by the rate of heating and sluggishness of melting. Silicate systems in general attain equilibrium too slowly for thermal analysis to give accurate information. The method is valuable in exploratory work but should otherwise be used with greater discretion than is customary.

The agreement obtained between the quenching data and the heated-stage microscope observations, where comparison is possible, is much closer than had been expected. Indeed more reliance is placed on the microscope determinations of the liquidus at concentrations of spinel greater than 30%. The starting materials for these mixes could not be prepared at temperatures high enough to obtain complete liquid. There was evidence to suggest that in some quenches the apparent precipitation of spinel was the result of segregation and retention of crystals in portions of the quench packet; these were drained of liquid as melting took place and never regained contact with the major part of the melt. This difficulty did not occur with the microscope observations, in which it was noted that the melt was very mobile and that a considerable amount of stirring was taking place. The quantity of material examined on the heated stage at any one time is only about 0·1 mg. The uniformity of any preparation therefore is subjected to a very critical test when such a small sample is taken. For some preparations several samples were taken for the microscope determinations as a precautionary measure. The very close agreement obtained between these individual results, and between the microscope and quench results as a whole, demonstrate the effectiveness of the procedure used to obtain homogeneity in making the initial preparations.

The heated-stage microscope, when skilfully used, could probably equal the accuracy of the quenching method, although its use at present has been limited to exploratory work. To expedite the initial exploration it would be valuable to have a more rapid method of preparing trial compositions. A ' wet ' method of preparation, such as that described by Roy,[14] would be ideal. In this method the required proportions of the component oxides of the system are mixed in the form of organic derivatives in alcoholic solution. The hydroxides are then co-precipitated by hydrolysis as a gel in which the components are intimately associated. This gel could be sampled by the heated thermocouple and the homogeneous mixture of hydroxides reacted together *in situ* to produce the melt to be studied. If the entire procedure proves to be practicable, the total saving in time and cost compared with conventional techniques would be of enormous benefit to high-temperature research.

Acknowledgments

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The Enthalpy of a 0·12% Carbon Steel

By J. R. Pattison, B.Sc., D.I.C., Ph.D., A.Inst.P., and T. H. Lonsdale

SYNOPSIS

A 0·12% carbon, 0·44% manganese, steel has been investigated by water calorimetry over the temperature range of 1000–1630° C. From the enthalpy data obtained, values have been assigned to the heats of transformation at the γ to $\delta + \gamma$ phase change temperature (1467° C), the peritectic temperature (1481° C), and the liquidus temperature (1509° C). The mean specific heat was found to be 0·157 in the austenite phase compared to 0·161 for pure iron, and 0·146 in the liquidus compared to 0·144 for pure iron.

1218

A KNOWLEDGE OF ENTHALPY for various commercial steels is necessary for the calculation of heat balances in steelmaking processes. In particular, enthalpy data are required for the determination of the heat content of a steel ingot during solidification in a mould. By conserving the heat contained in the ingots, an improvement in the fuel consumption of soaking pits can be obtained if an optimum cooling time for the ingot can be set before it is charged into the pit.

EXPERIMENTAL WORK

The method of determination of enthalpy was by mixtures using the water calorimeter and experimental procedure described previously.[1]

The steel investigated had the composition shown in Table I.

In work carried out to obtain the iron–carbon constitutional diagram, Adcock[2] used thoria crucibles to prevent any removal of carbon from the specimens by reaction with alumina crucibles. However, the free-energy/temperature diagrams published by Richardson and Jeffes[3] indicate that the reduction of alumina by carbon should not occur at temperatures below 2000° C. To justify the continued use of alumina crucibles in the investigations now reported, a specimen of the carbon steel under investigation was heated in a standard alumina crucible to 1520° C in an atmosphere of nitrogen and maintained at this temperature for four hours, which is much greater than the 20 minutes' time of soaking necessary before a calorimetric experiment. After cooling in the furnace, the sample was analysed and its composition was found to be unaltered by the furnace treatment. The original method of investigation, as used for pure iron, was therefore followed for the carbon steel, using alumina crucibles.

A thermal analysis of the steel was carried out to determine the temperatures of the allotropic changes and the arrest points shown in Table II were obtained.

These arrest temperatures indicate that the γ to $\delta + \gamma$ transformation occurred at 1467° C, and that the peritectic and liquidus temperatures were 1481° C and 1509° C respectively. These temperatures may be compared with the mean of the values derived from the iron–carbon diagrams of Ruer and Klesper,[4]

Table I
STEEL ANALYSIS

Carbon	0·12%
Silicon	<0·01%
Manganese	0·44%
Sulphur	0·022%
Phosphorus	0·035%
Nickel	0·08%
Chromium	0·03%
Copper	0·14%
Aluminium	<0·02%
Nitrogen	0·003%
Iron	balance

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Dr. Pattison and Mr. Lonsdale are at the Physics Department.

Table II
ARREST POINTS FOR 0·12% CARBON STEEL

	Falling Temperatures	Rising Temperatures	Falling Temperatures
Temperature of the γ to $\delta + \gamma$ change	1466·7° C	1466·8° C	1466·8° C
Peritectic temperature		1480·7° C	1480·8° C
Liquidus temperature		1509·0° C	1509·2° C

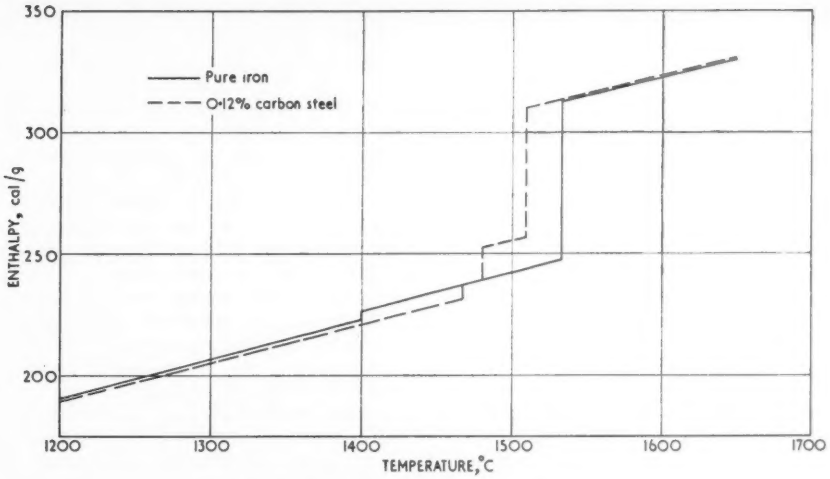

Fig. 1—Enthalpy of a 0·12% C Steel

Andrew and Binnie,[5] and Adcock,[2] corresponding to 0·12% carbon, namely 1462°, 1488°, and 1521° C. These values have been corrected to the 1948 International Temperature Scale. The iron–manganese diagram due to Gayler[6] shows that 0·44% manganese reduces the melting point of iron to 1514° C and raises the A_4 temperature to 1444° C; but a peritectic transformation does not exist for concentrations of less than 1% manganese. The other steel constituents, particularly phosphorus which Haughton[7] has shown to have a strong influence even at low concentrations on the transformation temperatures of iron, must also have their effects; and in the circumstances it is surprising that the transformation temperatures of this steel are so close to the values predicted by the iron–carbon diagram. One of the steels (*JW*) analysed by Andrew and Binnie[5] had a similar composition to that under investigation (0·13% C, 0·02% Si, 0·46% Mn, 0·025% S, 0·006% P, trace Ni, 0·01% Cr). The liquidus, peritectic, and γ to $\delta + \gamma$ transformations of this *JW* steel occurred at 1528° C, 1485° C, and 1462° C respectively. These figures are in fairly good agreement with those now reported except for the liquidus temperature which is surprisingly high.

In all 25 enthalpy measurements were carried out in a random order of temperature and the values obtained are given in Table III.

The enthalpy H_o^T at a temperature T is with respect to zero enthalpy at 0° C.

Table III

ENTHALPY RESULTS FOR 0·12% CARBON STEEL

Temperature T, °C	Enthalpy H_o^T, cal/g	
1007·6	159·70	γ-phase
1060·5	167·86	10 measurements
1114·6	175·39	
1182·7	186·42	
1213·7	191·98	
1254·5	198·47	
1319·4	207·97	
1362·2	214·84	
1408·9	221·91	
1451·3	229·38	
1472·1	238·00	$\delta + \gamma$ phase
1476·8	238·97	3 measurements
1479·1	239·09	
1483·2	252·90	δ-phase (liquid and
1492·5	254·31	solid state)
1499·6	255·44	3 measurements
1510·7	309·65	δ-phase (liquidus)
1523·1	312·12	9 measurements
1524·9	312·00	
1527·5	312·19	
1546·1	315·34	
1564·0	317·72	
1578·6	319·72	
1598·2	322·69	
1621·9	326·09	

Table IV

ENTHALPY OF A 0·12% CARBON STEEL

	Temperature, °C	Enthalpy H_o^T, cal/g	
	1000	158·2	
	1050	166·0	
	1100	173·8	
	1150	181·7	
	1200	189·5	
	1250	197·3	
	1300	205·2	
	1350	213·0	
	1400	220·9	
	1450	228·7	
γ to $\delta + \gamma$ change	1467	231·3	Heat of transformation 5·9
	1467	237·2	
Peritectic	1481	239·5	Heat of transformation 13·1
	1481	252·6	
Liquidus	1509	256·9	Heat of transformation 52·8
	1509	309·7	
	1550	315·7	
	1600	322·9	
	1650	330·2	

Regression lines computed with these results have the following equations with standard deviations σ.

1000° to 1467° C, γ-phase

$$H_o^T = 1{\cdot}47 + 156{\cdot}70\left(\frac{T}{1000}\right) \qquad \sigma = 0{\cdot}44$$

1467° to 1481° C, $\delta + \gamma$ phase

$$H_o^T = -2{\cdot}19 + 163{\cdot}20\left(\frac{T}{1000}\right) \qquad \sigma = 0{\cdot}19$$

1481° to 1509° C, δ-phase (liquid and solid state)

$$H_o^T = 23{\cdot}42 + 154{\cdot}71\left(\frac{T}{1000}\right) \qquad \sigma = 0{\cdot}02$$

1509° to 1650° C, δ-phase (liquidus)

$$H_o^T = 90{\cdot}02 + 145{\cdot}57\left(\frac{T}{1000}\right) \qquad \sigma = 0{\cdot}32$$

DISCUSSION

The regression lines obtained for the enthalpy of a 0·12% carbon steel over a range of temperatures are illustrated in Fig. 1, together with those previously determined for pure iron.[1] Smoothed values of enthalpy obtained from these equations for the steel are given in Table IV.

Over the temperature range investigated, the effects of the steelmaking additions to the pure iron on its enthalpy appear to be almost exclusively restricted to the 1400–1533° C range where modification of the allotropic changes occurs. At 1000° C the steel and iron have the same enthalpy values, and, although an increasing difference in enthalpy occurs with rising temperatures, the difference does not amount to 1% at 1400° C.

The change of state from γ-iron to molten δ-iron occurs in two stages involving 3·7 cal/g and 65·5 cal/g, so that a sum total heat of transformation of 69·2 cal/g is involved. The change of state of the carbon steel from the γ-phase to the δ-liquidus occurs in three stages involving 5·9, 13·1, and 52·8 cal/g. The sum total heat of transformation for the steel is thus 71·8 cal/g, so that the extra complexity of the transformation only involves an extra 2·6 cal/g. The difference in enthalpy above 1533° C between pure iron and 0·12% carbon steel is less than 0·1%.

There are no published enthalpy data for carbon steels above 1300° C, but the values now reported for 1000–1300° C may be compared by interpolation with those previously obtained by Umino[8] and by a number of investigators at the N.P.L.[9] which are illustrated graphically in a previous report.[10] Over this temperature range the figures now given are 1–2% lower than the N.P.L. values and 6–7% lower than the Umino values. Thus, a better agreement is obtained with the data obtained at the N.P.L. using an adiabatic calorimeter than with the water calorimetry results of Umino. However, the trend of lower enthalpy compared to pure iron is in more agreement with the simple curves based on Umino's data than the complex curves obtained by correlating the N.P.L. data.

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APPENDIX I

Enthalpy of Low-Carbon Steel between 0° and 100° C

The final calorimeter temperatures in the reported measurements of enthalpy varied between 35° and 40° C. To refer each individual determination to a common reference temperature at 0° C the enthalpy of steel at the final calorimeter temperature, referred to 0° C, must be added to the experimental value.

There are few published values for enthalpies of steels between 0° and 100° C but the N.P.L. data[9] at temperatures of 50–250° C for steels containing 0·06% to 0·4% carbon fit the following equation for enthalpy H_o^t at temperature t° C,

$$H_o^t = 11{\cdot}0\frac{(t)}{(100)} + 0{\cdot}4\frac{(t)^2}{(100)}$$

The change of enthalpy due to change of carbon content is apparently insignificant over this range.

This equation gives values for enthalpy between 35° and 40° which are less than 0·2 cal/g above the values for pure iron quoted in a previous report.[1] The work of Baerlecken[11] and Umino[8] confirms that low-carbon steel has a slightly greater enthalpy than pure iron at temperatures below the A_2 point, so this equation was used to refer the experimental values now reported to a 0° C datum temperature for enthalpy. This addition involved less than a 2% change in the experimentally determined values so that a small inaccuracy in the values given by the equation does not introduce an error of significance.

Determination of Low Carbon Contents in Steel

By the Methods of Analysis Committee

SYNOPSIS

The methods available for the determination of carbon in steels have been outlined and assessed with special reference to the determination in low-carbon steels, i.e. $< 0{\cdot}05\%$ carbon. The amount of carbon dioxide formed by combustion is a measure of the carbon content of the steel. Gravimetric, titrimetric, pneumatic, and conductimetric methods for the measurement of carbon dioxide are evaluated in detail. 1261

Part I: REVIEW OF METHODS AVAILABLE

THE DETERMINATION OF CARBON in steel almost invariably includes, as an essential operation, the combustion of carbon to carbon dioxide, and the amount of this gas so formed is the ultimate measure of the carbon content of the steel. Since carbon dioxide is a chemically active gas, methods for the determination of carbon in steel differ primarily in the means adopted for the measurement of a given amount of this gas. Thus a variety of methods is available which includes either a gravimetric, titrimetric, or pneumatic measurement of carbon dioxide or of some reaction product of carbon dioxide. The purpose of this report is to outline and assess some of the principles which have been applied to the measurement of carbon dioxide, and hence of carbon in steel, and to discuss their application to the special problem of low-carbon steels, i.e. $< 0{\cdot}05\%$ carbon, where greater accuracy is required. In this report, discussion of the metallurgical significance of these low carbon contents would be irrelevant, and their importance in special steels for the electrical and chemical industries need not be stressed.

Paper MG/D/32/55 of the Methods of Analysis Committee of the Metallurgy (General) Division of the British Iron and Steel Research Association, received 21st December, 1955.

The constitution of the Committee during the investigation was: Dr. E. GREGORY (*Chairman*), Edgar Allen and Co., Ltd.; Mr. N. F. ASTBURY (*left* 1949), G.K.N. Group Research Laboratories; Mr. B. BAGSHAWE, Brown-Firth Research Laboratories; Mr. R. BROWN (*joined* 1952), Morris Motors Ltd.; Mr. W. F. CHARTERIS, Colvilles Ltd.; Mr. S. W. CRAVEN, Alkali Division, I.C.I. Ltd.; Mr. F. H. EDWARDS (*joined* 1950), Bragg Laboratory, N.O.I.D.; Mr. W. T. ELWELL (1951-54), Billingham Division, I.C.I. Ltd.; Mr. R. F. JONES (1950–53, *rejoined* 1954), Steel Company of Wales Ltd.; Mr. D. MANTERFIELD (*joined* 1953), Steel, Peech and Tozer; Mr. G. MURFITT (*left* 1951), W. Jessop and Sons Ltd., later at Metals and Methods Ltd.; Mr. H. G. Short (*joined* 1949), National Physical Laboratory; Mr. G. E. SPEIGHT, United Steel Companies Ltd., later at R.T.S.C. Laboratories; Mr. S. D. STEELE, Babcock and Wilcox Ltd.; Mr. E. J. VAUGHAN (*left* 1950), Bragg Laboratory, N.O.I.D., later at R.N.S.S. H.Q.; Mr. T. R. WALKER (*left* 1950), English Steel Corporation Ltd.; Mr. W. WESTWOOD (*to* 1953, *replaced by* Mr. W. E. CLARKE, *left* 1954), British Cast Iron Research Association; Mr. T. H. WILLIAMS (*joined* 1954), Stewarts and Lloyds Ltd.; Mr. J. H. WRIGHT (*left* 1952), Dorman Long and Co., Ltd.; Mr. J. O. LAY (*Secretary*, *left* 1955), B.I.S.R.A.

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GRAVIMETRIC PROCESSES

Conventional Gravimetric Processes

The conventional method for the determination of carbon in steel is a gravimetric procedure based on the absorption of the carbon dioxide in a solid or liquid reagent. Solid absorbents are now customary and one form of this method, generally suitable for carbon contents to $\pm 0{\cdot}01\%$ reproducibility, has been adopted as a British Standard method.[1] When this method is applied to low-carbon steels, the 'blanks' which arise from several causes, e.g. boat, tube, or flux, and the difference-weighing errors due to the relatively large absorbent tube, assume a greater significance. This is sufficient to prevent the realization of third decimal place reproducibility and even to render the second decimal place of doubtful validity.

The advantages and simplicity of this method, however, have given impetus to the development of modifications capable of yielding greater accuracy. In a later section, a summary of work by Bagshawe and Pinder,[2] dealing with refinement of the standard combustion method for this purpose, is presented. The modifications are directed towards reducing the significance of the normal operational 'blank.' Thus, the use of a large combustion tube, which permits an increased sample weight of up to 6–8 times normal, and of an absorption tube of $\frac{1}{3}$–$\frac{1}{4}$ normal weight reduces the equivalent 'blank' and improves the reproducibility in the third decimal place. Special attention to the normal points of combustion carbon technique is required, e.g. elimination of oxides of sulphur, moisture content of combustion gases, and temperature variations of absorbent tube.

Carbon Separation Methods

Chemical separation of the carbon by solution of the sample in an appropriate solvent, followed by combustion in oxygen to carbon dioxide, is a long-established technique.[3] Since large weights of sample can be so treated, thus effecting a 'concentration' of

carbon, the principle may be used to advantage in the analysis of low-carbon steels. The usual solvent is copper potassium chloride or copper ammonium chloride, and on 27-g sample weights 0·001% carbon is equivalent to 0·001 g of carbon dioxide. The method should, therefore, be capable of third decimal place reproducibility.

This method has been examined at the National Physical Laboratory and applied in the co-operative investigation described in Part II. On high-purity irons and silicon irons of 0·01% carbon or less, satisfactory results have been obtained, but alloy steels, e.g. molybdenum and some nickel–chromium steels, have given low results. Experiments have shown that there is no loss of carbon during dissolution of the sample and it is believed that the low results arise from the formation of colloidal carbon which escapes separation on filtering the solution.

As the time requirements severely limit the application of this principle (solution of the sample may take up to 2 h) and, as other more practical procedures are now available for low carbon contents, further study has been discontinued.

PNEUMATIC PROCESSES

The amount of carbon dioxide formed from the combustion or carbon can be ascertained from its volume at constant pressure or from its pressure at constant volume. Both principles have been adapted to the determination of carbon in steel.

Volumetric Procedure

The volumetric measurement of the carbon dioxide evolved in the combustion of the sample forms the basis of the so-called 'Ströhlein' method for the determination of carbon. The exhaust gases from the combustion tube are scrubbed free from sulphur gases and collected in a gas burette, and their volume is measured. After passing through a solid or liquid carbon dioxide absorbent, the volume of the oxygen is measured. The difference between the two measured volumes is corrected according to the prevailing atmospheric pressure and temperature conditions. The corrected volume of carbon dioxide is then converted to carbon percentage on the original sample.

This method has been practised for many years and is extremely rapid; the inherent accuracy of the method is comparable, on low-carbon steels, with the conventional gravimetric procedure. It is not possible, however, by this method to obtain results which are comparable in accuracy with the pneumatic low-pressure method.

Low-Pressure Methods

Measurement of the pressure of carbon dioxide at a known volume is the basis of the 'low-pressure' method for the determination of low carbon contents. In this method, which was first described in 1920 by Yensen[4] and later applied and improved by Wooten,[5] Murray,[6] Naughton,[7] and others, the sample is heated by induction and burned in purified oxygen in a closed system. The apparatus is evacuated through a liquid-oxygen trap which condenses and retains the carbon dioxide whilst allowing the excess oxygen to escape; the solid carbon dioxide is then vaporized into a known volume and its pressure measured.

In the form described by these U.S. workers, the apparatus is expensive and complicated, and the method is long and hardly suitable for general laboratory conditions. However, the method has been considerably simplified by applying the techniques of the conventional combustion method, i.e. combustion of the sample in a stream of oxygen in a tube furnace, followed by absorption of the sulphur gases. Wells[8] has described a simplification of the method on the above lines and further improvements have been effected by other British workers,[9] so that in its present form the low-pressure method is almost as simple and as speedy in output as the conventional gravimetric combustion method. A summary of the latest form of the method, which is now in regular operation in this country, is given in Part III.

By close attention to operational details, i.e. purification of oxygen, blanks, etc., the low-pressure method as described in Part III is well able to give a reproducibility of $\pm$ 0·0005% carbon from 2·729 g of most low-carbon materials. With a proportionately reduced sample weight, the method may be applied to higher carbon materials.

TITRIMETRIC METHODS

The titrimetric methods involve the use of a liquid absorbent, usually a solution of barium hydroxide in water, and the determination is completed by titrating the excess alkali[10] or, less often, by filtering off the precipitated barium carbonate, redissolving, and titrating the resulting solution.

This principle has been adapted to the determination of low carbon contents, since it is claimed to eliminate many of the potential errors of the gravimetric estimation using solid absorbents, and with simple apparatus is capable of an inherently greater sensitivity than the standard gravimetric method. Accuracy can also be improved by the use of multiple factor-weight samples.

The method has, however, certain disadvantages which require special attention. Thus baryta solutions are relatively poor absorbers of carbon dioxide, particularly at high gas rates. The solution has to be prepared, stored, and used under conditions which preclude contamination with atmospheric carbon dioxide, and its nature makes accurate measurement of the volume somewhat difficult. The essential difference in the various forms of baryta-absorption titrimetric methods therefore lies in the special means adopted to limit the amount of baryta solution necessary, to achieve complete absorption of the carbon dioxide, and to ensure freedom from contamination. The methods described in Part III of this report are typical forms of this titrimetric method which have been practised by their sponsors with satisfaction for many years. Their experience and details of operation should be of value.

CONDUCTIMETRIC METHODS

The principle of the conductimetric method is the determination of the change in conductance of a barium hydroxide solution when used for the absorption of the carbon dioxide produced on combustion of the sample. It has been practised for medium-high carbon contents for many years,[11] and in recent years has been applied to the determination of low carbon

contents on account of its potentially greater accuracy than that possible by the standard gravimetric or titrimetric procedures.[12]

As the conductance is determined by a bridge method, it is the proportionate change rather than the absolute change which is of importance. The weaker the initial baryta solution, the greater the proportionate change; hence for low carbon contents, dilute baryta solution is essential. This factor must be balanced against the difficulty of obtaining complete absorption (*see* Titrimetric Methods) particularly from a rapid gas stream. Specially designed absorbers are necessary, therefore, and these should incorporate the electrodes for measurement of the conductance, and be fairly simple to dismantle and clean.

Since the amount of carbon dioxide evolved from the sample is small and the gas-flow restriction limits the weight of sample possible, careful attention is necessary to avoid contamination with atmospheric carbon dioxide both in the combustion operation and in the preparation and handling of the baryta solution. With careful attention to such detail, including temperature control of the absorber to 0·1° C, reproducibility of ± 0·0005% carbon is possible.

A short description of one form of this method is given in Part III of this report.

PART II: CO-OPERATIVE EXAMINATION OF SAMPLES OF LOW CARBON CONTENT

The co-operative investigation of methods for the determination of low carbon contents in irons and steels was commenced in 1948 by the Pig Iron, Carbon, and Low-Alloy Steels Analysis Sub-Committee and was subsequently extended to external collaborators under the ægis of the B.I.S.R.A. Methods of Analysis Committee.

PRELIMINARY EXAMINATION

It was realized at the outset that, whilst the existing standard method for carbon (B.S. 1121: Part 11: 1948) would readily give an accuracy of 0·01% carbon, the increasing demand for even closer limits, particularly in iron for electrical purposes and in highly alloyed steels, might necessitate a modified or even a completely different procedure. It was possible, however, that by close attention to detail, by increasing the sample weight, or by other modifications the normal accuracy of the standard combustion method might be improved. However, certain collaborators had already some experience of modified procedures, designed for higher accuracy with low carbon contents, of which the titrimetric procedures following absorption of carbon dioxide in baryta solution were most popular. Initially, therefore, samples were prepared for co-operative examination by the different procedures then in use by the co-operators, and the results of this preliminary work are summarized in Table I. In all cases the sample millings or drillings were degreased in ether before analysis and this was made standard practice throughout the subsequent work.

The results on the first sample MGS/54, a low-carbon steel, were unsatisfactory on account of its variable particle grading (this sample was a mixture of coarse and fine millings and the finer material appeared to be of higher carbon content than the average sample). The results on further circulated samples of low-carbon steel, MGS/69, 70, and 71, were more satisfactory and all co-operators were in agreement on the carbon contents of these three samples to the second decimal place, i.e. 0·02%, 0·01%, and 0·03% respectively. Comparing the results to third decimal place from the two main methods used, i.e. the combustion gravimetric and the combustion titrimetric methods, those from the latter appeared to be slightly lower and more consistent. The results from the less frequently used methods, i.e. combustion following decomposition in copper ammonium chloride, the volumetric Ströhlein procedure, and the conductimetric methods were in each case in good agreement with mean values by the combustion-titrimetric method.

In general these results were very gratifying; they gave promise of ultimately achieving third decimal place accuracy by conventional techniques readily applicable in works analytical laboratories and thereby of avoiding the necessity for the specialized techniques which had been developed elsewhere for this problem of low carbon contents.

GENERAL EXAMINATION

At this stage the investigation was extended to include alloy steels and certain external collaborators having special experience of the low-carbon problem were invited to assist. Four new samples were prepared:

MGS/91—A basic Bessemer heat deliberately blown to produce an abnormally low carbon content and subsequently killed with aluminium
MGS/92—A low-carbon basic O.H. heat
MGS/93—An 18/8 Cr–Ni heat
MGS/94—A 14/9 Cr–Ni heat.

The two latter materials were 18-lb experimental high-frequency heats. A large weight of carefully prepared millings was prepared from each material, mixed, and identical portions distributed to the collaborators.

The results are classified and summarized in Tables II*a–e*, each of which relates to a separate method or modification. In most cases more than two determinations have been made on each sample, but for simplification only the mean and the upper and lower limits have been included. In addition to the method used in the preliminary examination, several results by the combustion gravimetric method using a multiple factor-weight sample are available.

COMMENTS ON RESULTS AND METHODS

From a critical assessment of the various results and of the inherent accuracy of the different procedures, the carbon contents of the four samples may be taken as:

MGS/91—$0{\cdot}010_5$%
MGS/92—$0{\cdot}032_5$%
MGS/93—0·024%
MGS/94—$0{\cdot}036_5$%

These values serve, therefore, as a basis for comparison of the various methods and results.

Table I

PRELIMINARY RESULTS ON LOW-CARBON SAMPLES

Analyst	Sample Weight, g	Temp., °C	Flux	Remarks	MGS/54	MGS/69	MGS/70	MGS/71
				Barium Hydroxide Volumetric Method				
1	5	1050–1100	2g Powdered magnesite + 2g red lead	Solutions standardized against low-C steel	0·024 (0·022–0·025)	0·0219 (0·0215–0·0223)	0·0084 (0·0079–0·0087)	0·0281 (0·0277–0·0285)
	10						0·0085, 0·009	0·029, 0·030
2	5	1100–1150				0·022 (0·018–0·024)	0·010 (0·009–0·012)	0·029 (0·028–0·030)
3	2	1150	2g Lead foil	*Analyst*, 1945, vol. 70, p. 466 $BaCl_4$–NaOH absorption in vacuo, potassium acid phthalate titrant		0·024, 0·024	0·009, 0·012	0·034–0·033
				Copper Ammonium Chloride Decomposition				
3	10	1100	None	Coarse particles (MGS/54)	0·016, 0·016 0·016	0·021, 0·024	0·010, 0·012	0·030, 0·031
4	5			Fine particles 10-mesh (MGS/54)	0·023, 0·024			
	10					0·0245	0·010, 0·011	0·034, 0·034
				Gravimetric Finish				
4	3			Fine particles MGS/54	0·022			
	5			—10-mesh	0·019	0·024, 0·024	0·011, 0·012	0·035, 0·035
5	4	1100	Red lead	Coarse 20-mesh MGS/54	0·028	0·023		0·037
				Fine MGS/54	0·033			
	4		Lead	Coarse 20-mesh MGS/54	0·030	0·022		0·034
				Fine MGS/54	0·034			
	4		Lead chromate		...	0·022		0·035
6	5	1150			0·028, 0·029	0·023	0·011, 0·013	0·037
7	F			Coarse MGS/54	0·014	0·021, 0·019		
	4	1050	Red lead	Fine MGS/54	0·025–0·035*	0·020, 0·018		
	2F					0·017, 0·018		
2	2				0·025			
	5				0·025			
	2		Red lead		0·029			
8			Red lead	Coarse MGS/54	0·014			
				Fine MGS/54	0·025–0·035*			
				Ströhlein Procedure				
4	5			Fine particles—10-mesh (MGS/54)	0·019, 0·021	0·0225 (0·022–0·023)	0·0115 (0·011–0·012)	0·032, 0·033
2	2	1200–50		O_2 75 ml/min	0·025			
	5	1200–50			0·025			
	2	1200–50	Red lead		0·029			
	5	1200–50		Average of 3 tests		0·020	0·010	0·032
				Conductimetric Finish				
9	2·5	1250		O_2 flow 700 ml/min	...	0·022 ± 0·001	0·008 ± 0·001	0·035 ± 0·001

* Figs. variable

Combustion Gravimetric Method

Although in general the mean values by the single factor-weight combustion method are slightly higher, they are in surprisingly good agreement in the third decimal place with the accepted values shown above. It is to be expected that this agreement arises from the averaging of many replicate tests, as the spread of results by each collaborator is wider than would warrant third place reproducibility from a single test.

Results by the multiple factor-weight combustion method described in Part III are in excellent agreement with the accepted values and have good reproducibility within each laboratory. Certain co-operators show minor tendencies in one direction and this may be caused by slight error in assessing the true blank. Under careful conditions, however, this method fulfils the need for a low-carbon method capable of third decimal place reproducibility.

Decomposition in Copper Ammonium Chloride followed by Combustion

This method has the attraction of being able to deal with a large weight of sample. It is, however, time-consuming and unsuitable for routine repetition work. On the two unalloyed samples it has given accurate results, but has clearly failed on the two high-chromium samples. Although its application is limited, its value as an occasional alternative for special samples should not be dismissed.

Table IIa

GRAVIMETRIC COMBUSTION METHOD

Analyst	Operation Details			Carbon Steels		Cr–Ni Stainless Steels		Notes
	Sample wt, g	Combustion Temp., °C	Flux Used	MGS/91	MGS/92	MGS/93	MGS/94	
Single Factor Weight (F)								
A	F	1150	Tin (2·7g)	...	...	0·037 (0·036–0·039)	0·040 (0·039–0·040)	Blank on calcined boat + flux = 0·0003g CO_2
B	F	1200	Lead (1·3g) tin (1·0g) or proportionate mixtures thereof	0·014 (0·013–0·015)	0·034 (0·030–0·038)	0·025 (0·023–0·027)	0·036 (0·033–0·038)	Blanks Pb foil 0·0009g CO_2 Sn 0·0003g CO_2
C	F	1000–1050	Tin (0·2g)	0·010 (0·008–0·012)	0·034 (0·032–0·036)	...	...	
		1150–1200	Tin (1·5g)	...	...	0·025 (0·024–0·026)	0·039 (0·036–0·042)	
D	F	1100–1150	Armco iron + tin shot	0·011	0·025	0·033	0·040	
E	F	1350	...	0·009 (0·008–0·011)	0·035 (0·034–0·036)	0·023 (0·019–0·027)	0·040 (0·039–0·041)	
Double Factor Weight (2F)								
F	2F	1150	Red lead (3g)	0·011 (0·010–0·012)	0·031 (0·031–0·031)	0·024 (0·023–0·024)	0·036 (0·035–0·036)	Blank ≡ 0·0004g CO_2
G	2F	1130	Lead	0·012 (0·011–0·013)	0·032 (0·030–0·033)	0·023 (0·022–0·023)	0·035 (0·035–0·036)	Blank ≡ 0·0009g CO_2
H	5·0	1150	Tin (1g)	0·013	0·032	0·031	0·035	...
Multiple Factor Weight								
A	4F	1050	Tin powder (10·91g)	0·012 (0·011–0·012)	0·034 (0·034–0·034)	0·037 (0·036–0·038)	0·043 (0·043–0·044)	Four boats contain flux and sample burned off in succession. Total blank ≡ 0·0012g CO_2
B	4F, 5F or 6F	1160–1200	Various weights of lead, tin, red lead, or mixtures of red lead and nickel	0·009 (0·009–0·010)	0·031 (0·031–0·032)	0·024 (0·024–0·025)	0·037 (0·036–0·037)	Blanks 0·0006–0·0014 CO_2
F	4F	1200–1250	Preheated red lead (7g)	0·010 (0·010–0·011)	0·034 (0·032–0·034)	0·025 (0·024–0·025)	0·037 (0·032–0·039)	Blank ≡ 0·0007g
H	20g	1050	Tin (10g)	0·013 (0·013–0·014)	0·035 (0·035–0·036)	0·026 (0·025–0·026)	0·038 (0·037–0·038)	...

Table II*b*

CHEMICAL DECOMPOSITION IN COPPER AMMONIUM CHLORIDE

Analyst	Operation Details			Carbon Steels		Cr–Ni Stainless Steels		Notes
	Sample wt, g	Combustion Temp., °C	Flux Used	MGS/91	MGS/92	MGS/93	MGS/94	
J	12–25g	1100	...	0·010 (0·010–0·0105)	0·035 (0·034–0·036)	0·017 (0·016–0·017)	0·030 (0·029–0·031)	Blank = 0·0004g CO_2, combustion gravimetric finish
K	5–10g	1100	...	0·010 (0·010–0·010)	0·032 (0·032–0·033)	0·019 (0·019–0·020)	0·030 (0·028–0·031)	Combustion with titrimetric finish

Table II*c*

LOW-PRESSURE PNEUMATIC METHOD

Analyst	Operation Details			Carbon Steels		Cr–Ni Stainless Steels		Notes
	Sample wt, g	Combustion Temp., °C	Flux Used	MGS/91	MGS/92	MGS/93	MGS/94	
L	2·0	1200	Lead (0·75g)	0·0088 (0·0087–0·0089)	0·0230 (0·0318–0·0321)	0·0235 (0·0231–0·0237)	0·0351 (0·0348–0·0353)	Blank = 0·0013% C
G	F*	1250	2g Red lead (pre-ignited)	0·0084 (0·0078–0·0091)	0·0319 (0·0318–0·0320)	0·0232 (0·0230–0·0232)	0·0361 (0·0360–0·0361)	Blank = 0·0002% C
M	2·0	1100	Lead-foil (1·0g)	0·0111 (0·0107–0·0115)	0·0321 (0·0320–0·0325)	0·0222 (0·0219–0·0225)	0·0356 (0·0354–0·0358)	Blank = 0·0028% C
J	2·0	1150	None	0·0099 (0·0093–0·0105)	0·0337 (0·0335–0·0340)	0·0223 (0·0220–0·0225)	0·0360	Blank = 0·0018% C

* Single Factor weight

Table II*d*

TITRIMETRIC METHOD

Analyst	Operation Details			Carbon Steels		Cr–Ni Stainless Steels		Notes
	Sample wt, g	Combustion Temp., °C	Flux Used	MGS/91	MGS/92	MGS/93	MGS/94	
F	5g	1200	2g Red lead	0·011 (0·011–0·012)	0·034 (0·033–0·034)	0·024 (0·023–0·024)	0·037 (0·037–0·038)	
K	2g	1150	2g Lead	0·010 (0·010–0·011)	0·033 (0·033–0·034)	0·025 (0·024–0·027)	0·038 (0·037–0·039)	
N	5–10g	1140–1190	Red lead and magnesite bedding—pre-ignited	0·013 (0·012–0·013)	0·033 (0·032–0·037)	0·027 (0·025–0·027)	0·040 (0·038–0·040)	Blank nil to 0·0003g CO_2
O	5g	1150–1200	0·5g lead + 0·5g tin	0·014 (0·013–0·015)	0·032 (0·031–0·033)	0·024 (0·023–0·025)	0·038 (0·036–0·039)	Blank 0·0006g CO_2
P	3–5g	1350	2g PbO 1g CuO	0·011 (0·011)	0·033 (0·032–0·033)		0·038 (0·038	
Q	2·5g	1250	...	0·013 (0·012–0·014)	0·034 (0·034–0·035)	0·019 (0·018–0·019)	0·036 (0·036)	No blank
R	5g	1050–1100	2g Red lead	0·010 (0·009–0·010)	0·034 (0·033–0·034)	0·022 (0·021–0·023)	0·038 (0·037–0·038)	Blank negligible

Table IIe

CONDUCTIMETRIC METHOD

Analyst	Operation Details			Carbon Steels		Cr–Ni Stainless Steels		Notes
	Sample wt, g	Combustion Temp., °C	Flux Used	MGS/91	MGS/92	MGS/93	MGS/94	
D	1g	1375–1425	Tin powder	0·011	0·030	0·025	0·036	
E	1g	1350	...	0·0084 (0·0083–0·0085)	0·0346 (0·0345–0·0348)	0·0243 (0·0235–0·0260)	0·0402 (0·0395–0·0410)	
S	1g	1150–1300	0·7g pre-ignited lead	0·0082 (0·0079–0·0085)	0·032 (0·0315–0·0326)	0·0222 (0·0221–0·0224)	0·0352 (0·0349–0·0356)	Blank $\equiv$ 0·00003g CO_2

Titrimetric Method

Modification of operational detail in the various laboratories is considerably greater in this procedure than in any of the other method principles examined. Nevertheless the results obtained are in quite good agreement.

Combustion Low-Pressure Pneumatic Method

This procedure appears to be capable of producing results of a high order of reproducibility and has the merit that a minimum of initial preparation is required to ensure satisfactory operation when the apparatus is used intermittently. It is, however, unlikely to have much appeal for the works routine analyst in view of the rather extended operational time, provided that results of sufficient accuracy can be obtained by an alternative method.

Conductimetric Method

The values obtained by this method indicate very good reproducibility. The agreement between the conductimetric figures obtained by Chirnside[12] and the low-pressure figures obtained by Wells and Speight are indicative of a new era in precision carbon determinations on low-carbon steels.

GENERAL COMMENTS AND CONCLUSIONS

This collaborative examination has shown that there are at least four procedures which will satisfy the basic requirements of accuracy, reasonable speed, and adaptability to general laboratory conditions for the determination of low carbon contents in irons and steels. These are:

(i) The modified combustion method using a multiple factor-weight sample
(ii) The barium hydroxide absorption-titrimetric method
(iii) The low-pressure pneumatic method
(iv) The conductimetric method.

For carbon contents below 0·05%, results in different laboratories by methods (i) and (ii) should be within 0·002% C, and by methods (iii) and (iv) within 0·001% C of the true content. Indeed, the measurement principle used in the two latter methods permits a considerably higher degree of reproducibility.

The four methods will each have a particular field of application. Methods (i) and (ii) will be favoured by the more routine laboratories where results to 0·002% C are normally adequate, since the techniques involved are more familiar to the works analyst. Methods (iii) and (iv) may be regarded as specialized refinements of technique which will be favoured more for research conditions and for investigations or problems which demand the highest accuracy of carbon determination. Another advantage arising out of the greater accuracy of methods (iii) and (iv) is their application to higher carbon steels when the amount of sample available is limited.

PART III: OPERATING DETAILS OF THE MOST USEFUL PROCEDURES

It has been shown in Part II that there are four main method principles capable of producing carbon values of sufficient accuracy to meet the most rigid commercial specification for carbon contents of low-carbon steels, without the introduction of expensive, time-consuming, or complex operational techniques.

These method principles have now all been in operation for several years, both as routine control procedures and as means of obtaining the ultimate degree of accuracy possible for special purposes.

The four papers comprising Part III of this report have each been summarized from information supplied by chemists having specialized knowledge and experience of the particular method. It will be appreciated that each method principle can be applied with considerable variation in operational detail, although each description has been confined in the main to a single version in current use. Whether the procedures as written are in the best possible form may be subject to question; it is certain, however, that each of the methods reported has already given a long period of satisfactory service.

MULTIPLE FACTOR-WEIGHT COMBUSTION METHOD[2]

Introduction

To meet the increasing demand for carbon determinations accurate and reproducible to the third decimal place, the orthodox combustion procedure has been modified to permit the combustion of much larger charges than have previously been used. The modified procedure described can be operated on

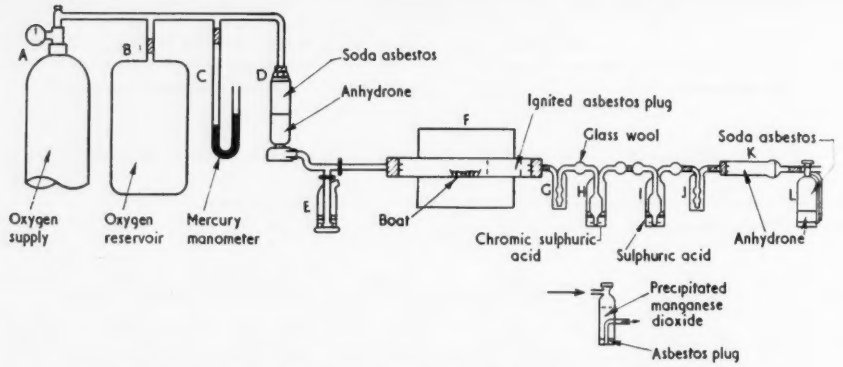


Fig. 1—Apparatus for the determination of carbon by combustion

several multiples of the normal factor weight, e.g. $6 \times 2{\cdot}729 = 16{\cdot}37$ g, and by this means the normal tolerance of $\pm$ 0·01% for the conventional process has been reduced to about $\pm$ 0·002%.

As the procedure is an adaptation of the normal process, it is particularly well suited to general production control in works routine laboratories and in this respect it appears to hold a marked advantage over alternative procedures, e.g. low-pressure measurement, which are more suitable for special and research applications.

The operational time is similar to that of the normal process, e.g. an output of five determinations per tube per hour can be maintained on a day by day basis. Complete combustion of practically all classes of low-carbon iron and steel can be effected by applying the conditions specified. The process has been found particularly effective for the austenitic and stainless ranges of low-carbon materials but it is not so well suited to meet ultimate accuracy requirements on materials such as 4% silicon transformer iron, where the critical effect of carbon has led to demands for fourth decimal place control.

Apparatus

The layout and general design of the apparatus corresponds to that described in B.S. 1121: Part 11: 1948. A diagrammatic representation is given in Fig. 1 and a photograph in Fig. 2. The following minor modifications are recommended:

(i) The oxygen supply passes through a refractory tube containing platinized asbestos at 700° C before the removal of carbon dioxide and water vapour by the soda asbestos and Anhydrone absorbents

(ii) The furnace (inside tube temperature 1200–1250° C) is of conventional design but has an aperture large enough to accommodate 2 in. outside dia. combustion tubes. Windings of heavy gauge Kanthal (Grade A1, 16 s.w.g.) or platinum are a useful alternative to radiation heating

(iii) The combustion tubes ($1\frac{1}{2}$ in. inside dia.) and boats ($4\frac{3}{4}$ in. × $1\frac{1}{4}$ in. × $\frac{5}{8}$ in. deep) are of high-grade aluminous refractory pre-ignited at 1200° C before use. The residual blank on a boat after pre-ignition should be less than 0·4 mg of carbon dioxide

(iv) In the absorption system large Arnold bulbs ($5\frac{1}{2}$ in. × $1\frac{1}{2}$ in. dia.) of chromic-sulphuric acid are used to avoid frequent recharging. Alternatively, precipitated manganese dioxide may be employed provided that a bubbler tube is incorporated in the train as a gas-flow indicator

(v) The carbon dioxide absorption tube is a miniature Midvale pattern bulb (4 in. × $\frac{3}{4}$ in.), weighing 30–35 g when packed. A 'calcium chloride' tube (4 in. × $\frac{1}{2}$ in.) weighing about 20 g when packed, may be advantageous for very low carbon determinations.

Method of Operation

(1) Sample weights up to 8 × factor weight (21·832 g) may be employed, but above 6 × factor weight combustion conditions are extremely severe; for pure iron and 4% silicon iron not more than 4 × factor weight is advisable, while for low-carbon ferrochromium a mixture of 8·187 g of sample with an equal weight of low-carbon iron is recommended.

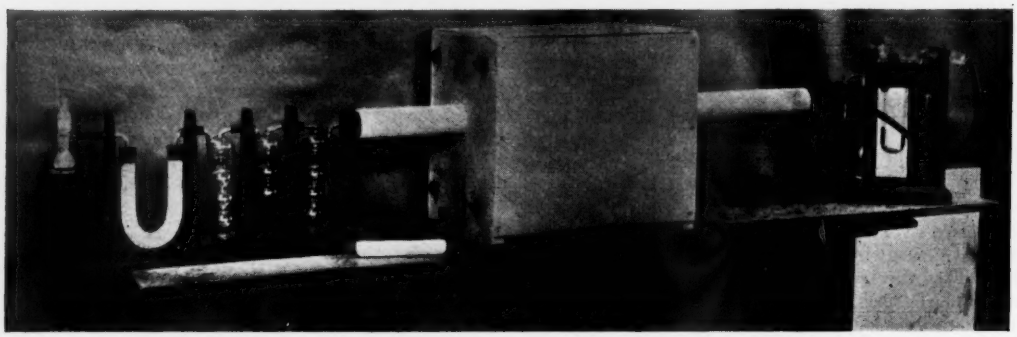


Fig. 2—Apparatus for the determination of carbon by combustion

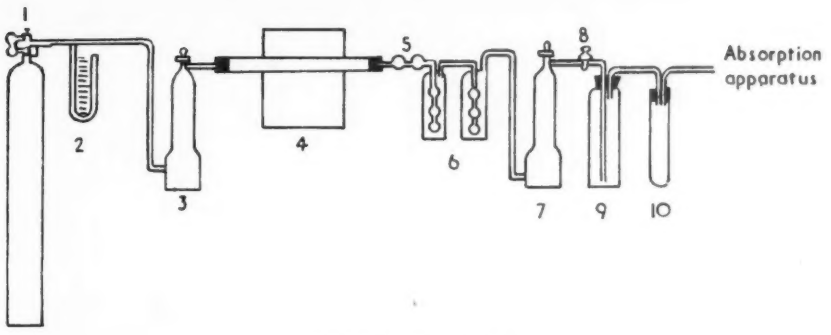


Fig. 3—Combustion train

The increase in weight of the absorption tube should not exceed 0·04 g per determination.

Sample preparation should result in short, fine, and dense pieces, coarse brittle materials such as ferro-alloys being crushed to pass 30-mesh B.S.S.

(2) A flux of 7 g of red lead, pre-heated at 450° C to give a residual blank less than 0·3 mg of carbon dioxide, should be used as a cover for each multiple factor-weight charge.

(3) The normal oxygen flow needs to be increased to between 1½ and 2 l./min for multiple factor-weight combustion, and manual or other means of ensuring a positive gas pressure throughout the combustion period should be employed.

(4) An internal tube temperature of 1200–1250° C is satisfactory for the combustion of most materials but 1100–1150° C is adequate for highly reactive materials such as pure iron and silicon iron. Complete combustion and removal of all carbon dioxide should be achieved within a total time of 10 min.

(5) The total blank correction is about 0·7 mg and seldom varies sufficiently to influence the third decimal place, e.g. on a 6 × factor weight basis the blank is equivalent to 0·0012% of carbon and an error of 0·1 mg therefore corresponds to only ± 0·0002% of carbon.

BARIUM HYDROXIDE ABSORPTION-TITRIMETRIC METHOD[10]

Introduction

In the determination of carbon in processed electrical sheet having carbon contents of 0·01% and under, with an accuracy approaching ± 0·0005% C, the use of large-weight gravimetric estimations is open to suspicion, both as to complete combustion of this bulk of metal and to the critical conditions necessary to control the accurate weighing of a heavy absorption bulb.

One alternative examined was a barium hydroxide absorption method, in which the gas was passed through the solution contained in a large Mayer tube and the precipitated carbonate filtered on a sintered glass filter and washed free from the absorption solution with hot water. The carbon dioxide was then determined by titration with standard acid, or by dissolving the precipitate in hydrochloric acid, precipitating the barium as sulphate and finishing gravimetrically.

Atmospheric contamination affected the reproducibility of this procedure, however, and this led to the consideration of a barium hydroxide absorption method in a closed circuit with a titration finish. A

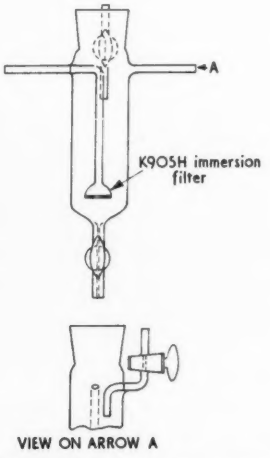


Fig. 4—Modification of absorption vessel (11)

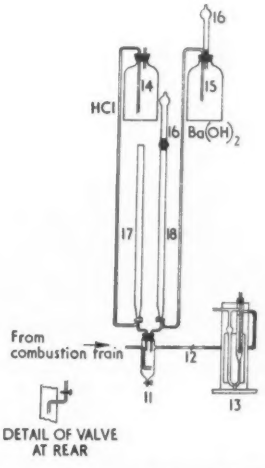


Fig. 5—Complete absorptiometric apparatus

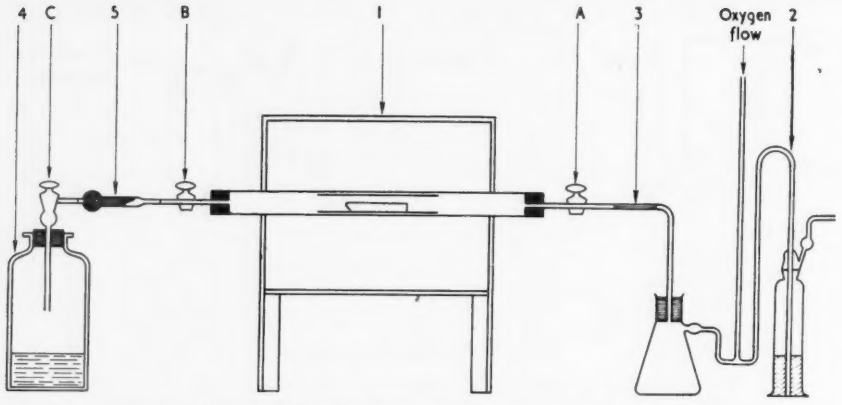


Fig. 6—Sodium hydroxide–barium chloride titrimetric method

method based on this principle was being operated in Australia; after some modifications it has been used successfully in this country over the last ten years.

Apparatus

The apparatus is illustrated in Figs. 3–5:

1. Oxygen supply, from cylinder with fine adjustment regulator.
2. Mercury pressure gauge to maintain 3 in. Hg.
3. Large drying tower charged with soda asbestos to remove CO_2.
4. Silit-rod electric furnace fitted with a Morgan ΔH combustion tube maintained at 1050–1100° C.
5. A two-bulb reduction tube packed with glass wool to retain volatilized iron and dust.
6. Two Dufty absorption bulbs; the first acting as a trap and the second containing chromic acid in water (a saturated solution diluted with an equal volume of water).
7. Calcium chloride tower containing $CaCl_2$ or Anhydrone.
8. Regulating stop-cock to build up a pressure (3 in. Hg) in the combustion tube.
9. Tube packed with zinc shavings to form a sulphur trioxide scrubber.
10. 5 in. × 1 in. test-tube safety trap to protect the train against any suction on the absorption apparatus.
11. Absorption apparatus.
12. 3-way stop-cock.
13. White-Wright flow meter.
14. Aspirator containing hydrochloric acid solution.
15. Aspirator containing barium hydroxide solution.
16. CO_2 trap for barium hydroxide.
17. Hydrochloric acid burette.
18. Barium hydroxide burette.

Reagents

Barium Hydroxide Solution—10 g of barium hydroxide ($Ba(OH)_2.8H_2O$) are dissolved in boiled distilled water, filtered, and cooled under a CO_2 trap. When cold, 2 mg of thymol blue indicator are added and the solution is diluted to 1 l. with boiled distilled water.

Standard Hydrochloric Acid—6·5 ml of hydrochloric acid (sp.gr. 1·16) are diluted to 1 l. and the solution is adjusted to the same strength as the barium hydroxide solution.

Method of Operation

(1) Cut the flow meter out of circuit, by turning the 3-way cock (12) to the atmosphere and run 30 ml of the standard $Ba(OH)_2$ solution into the absorption vessel.

(2) Replace the flow meter in circuit by means of the 3-way cock.

(3) Regulate the flow of oxygen by means of the fine adjustment to pass 130–140 ml/min; this is accomplished by first setting the tap (8) to half-open and keeping it in this position, then adjusting the oxygen valve and the flow meter until gauge (2) reads 3 in. and the flow meter 2½ in. Maintain these two readings by means of the fine adjusting valve on the oxygen cylinder throughout the whole 15 min required for the estimation.

(4) Take a boat (which has been previously heated until free from blank), weigh 2 g of powdered magnesite (90-mesh, calcined free from blank), and spread along the bottom of the boat. Weigh 5 g of the steel (for carbon of 0·04% and under) into the boat and sprinkle with 2 g of red lead, previously heated until free from blank. Place the boat and contents in the combustion tube.

(5) Regulate the flow of oxygen by means of the fine regulator, to allow for the extra amount used for burning the sample, still maintaining the two gauges at 3 in. and 2½ in. respectively. After 15 min open the 3-way cock to the atmosphere.

(6) Titrate with standard hydrochloric acid from a blue to a yellow colour as rapidly as possible, allowing the oxygen to pass all the time, thereby causing sufficient agitation. Never at any time shut off the oxygen during a run.

(7) Expel the waste liquid from the absorption vessel by opening the waste-cock and closing the 3-way cock.

(8) Close the waste-cock and immediately turn the 3-way cock to the atmosphere. The apparatus is now ready for the next determination.

Standardization and Calculation

Standardize the solution against a low-carbon steel under the same conditions as the test.

The solution is also checked against N/10 Na_2CO_3 and the theoretical carbon value determined:

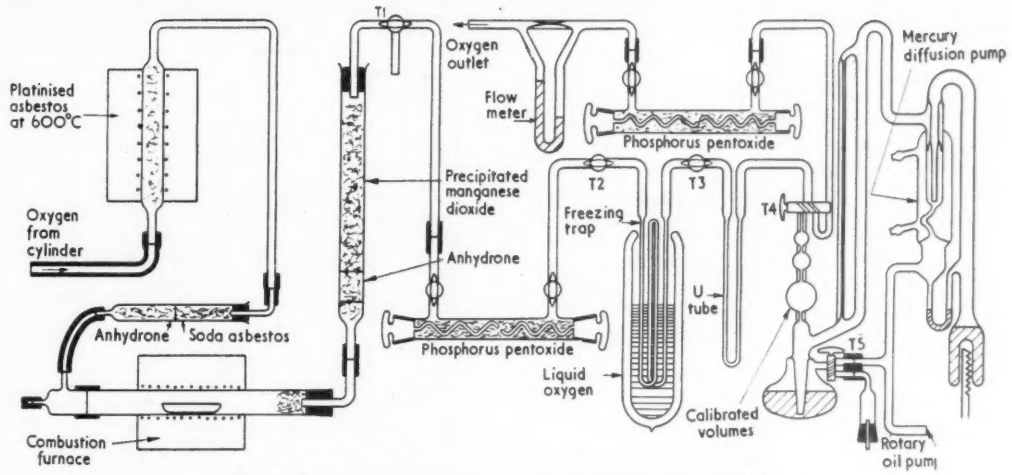


Fig. 7—Low-pressure apparatus for carbon determination

$$(1\ \text{ml}\ \frac{N}{10}\ Na_2CO_3 = 0{\cdot}0006\ \text{g C})$$

The actual and the theoretical carbon values should agree closely. In practice, standardization against a standard steel gave a carbon value of 0·00036 g C per ml compared with a theoretical value of 0·000363 g C per ml.

Sodium-Hydroxide–Barium-Chloride Modification

A slightly different procedure has been described by Cotton[13] and this has also given satisfactory service over a similar period. The main details of the apparatus (see Fig. 6) are as follows:

(i) *Furnace* operating at a minimum of 1000° C, increased to 1100° C for highly alloyed steels, recorded by a platinum–rhodium thermocouple and millivoltmeter
(ii) *Constant-pressure device* with safety trap
(iii) *Capillary tube* passing ¼ l./min of gas at atmospheric pressure
(iv) *Absorption bottle* 1 l. capacity, evacuated
(v) *Sulphur dioxide absorption tube* containing dry precipitated manganese dioxide
A and *B Control taps* for isolating the combustion tube, which is fitted with a protective pure nickel inner lining.

The absorbent used is a sodium-hydroxide–barium-chloride mixture (35 ml of 0·5 M barium chloride with a quantity of standard carbonate-free sodium hydroxide solution appropriate to the carbon content being measured). After absorption of the carbon dioxide, excess alkali is titrated with standard hydrochloric acid or potassium acid phthalate solution, using phenolphthalein indicator. Combustion of the sample is assisted by a 2-g strip of lead foil.

LOW-PRESSURE PNEUMATIC METHOD[9]

Introduction

The development of silicon–iron alloys and low-carbon stainless steels in recent years has necessitated more precise methods for carbon determination. Whilst the normal combustion method employing a weighed absorption tube is generally satisfactory at the higher carbon levels, it suffers serious limitations when applied to low carbon contents. Some improvement in accuracy is possible by the adoption of a semi-micro weighing technique, but for precise analysis the method is considered to be unsatisfactory.

Review of the published work on the determination of carbon in recent years has led to the conclusion that, of the various methods available, the low-pressure method is potentially the one of greatest precision.

The procedure recently adopted by Wells[8] showed that results of great precision are obtainable with comparatively simple apparatus and the present method merely attempts to introduce certain further modifications for ease of operation.

Apparatus

The complete apparatus is illustrated in Fig. 7. The details of the combustion procedure vary little from conventional technique, e.g. oxygen purified by absorption of carbon dioxide and water vapour after passage over heated platinized asbestos, and furnace of normal design, incorporating an asbestos wool filter. The liquid oxygen freezing trap retains the evolved carbon dioxide, which is subsequently measured by means of the calibrated volumes in the vacuum measuring system, covering a range of carbon contents up to 0·2% on a 2·729 g sample.

Method of Operation

In addition to the usual precautions for obtaining virtually blank-free boats and flux, samples are washed in ether and dried before weighing. The sequence of operations is as follows:

(i) Place an empty boat in furnace tube, pass oxygen for 2–3 min, remove the boat, and allow to cool. Weigh 2·729 g of sample, mix with 2 g of red lead and transfer to the boat. Insert into the mouth of the tube and pass oxygen for 2 min to sweep out entrained air, by-passing the stream of gas through T_1
(ii) Return T_1 to the normal position, open T_2, T_3, and T_4, and adjust the oxygen flow to 750 ml/min. The system from T_4 is maintained under vacuum by the pumps
(iii) Immerse the freezing trap, transfer the boat (by means of a steel rod inserted through the atmosphere trap) to the hot zone and allow combustion to proceed for 4 min, temporarily adjusting the oxygen flow if necessary to maintain a positive pressure

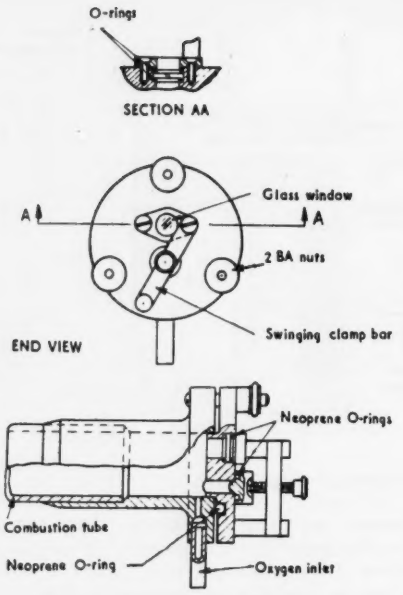


Fig. 8—Combustion-tube closure

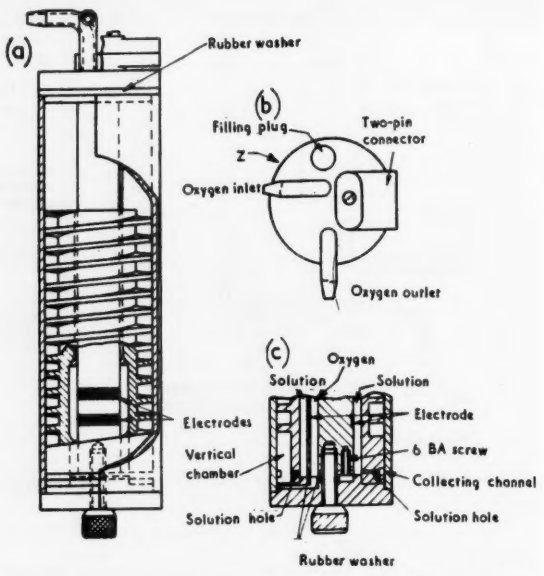


Fig. 9—Conductimetric method for carbon: conductivity cell showing (*a*) sectional view, (*b*) plan view, and (*c*) part section in direction *Z*, showing routes taken by the gas and by the solution into the base of the helix

(iv) By-pass the oxygen through T_1, close T_2, and slowly turn T_4 to connect with the calibrated bulbs. Replace the used boat by a new empty boat

(v) After 1 min rotate T_4 through 90°, transfer the liquid oxygen container to the **U**-tube, and warm the freezing trap to transfer the carbon dioxide to the **U**-tube. Prepare the next sample for combustion

(vi) Close T_3, open T_4, and open T_5 slowly to the air until mercury reaches the top calibration marks. Remove the liquid oxygen container from the **U**-tube and warm the latter gently (e.g. alternately hot and cold air from a hair drier). The gas should be transferred within about 1 min, when the mercury is raised to the highest attainable calibration point and the height in the measuring limb above this point is measured (on a fixed scale). The reading is corrected for the previously determined vacuum reading and the volume of carbon dioxide calculated at N.T.P.

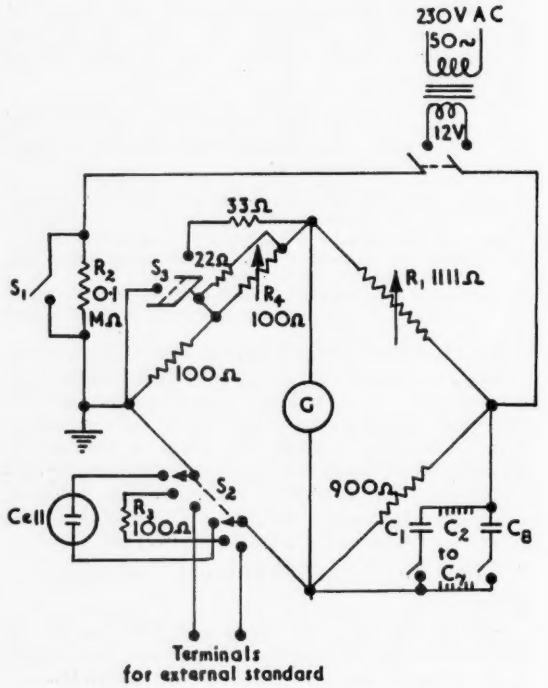
(vii) By careful manipulation of taps T_4 and T_5, expel the carbon dioxide from the apparatus through the diffusion pump and, after re-attaining vacuum conditions, close T_4. Readmit the oxygen flow to the freezing system via taps T_1, T_2, T_3 and T_4 and proceed as above to the next determination.

CONDUCTIMETRIC METHOD[18]

Introduction

Attention has already been focused on the need, in the steel industry, for the determination of small percentages of carbon because of the stringent requirements of the electrical engineer for silicon steel of low carbon content. Two main techniques have been developed: that known as the 'low-pressure' method and that involving the use of a large sample, say 20 g, for the combustion and subsequent weighing of the carbon dioxide on the usual solid absorbent.

In the low-pressure method, the carbon dioxide produced from the combustion of 0·5–2 g of sample is condensed in a liquid oxygen trap; it is then expanded into a previously evacuated vessel of known volume and its pressure is measured. Modifications



$C_1 = 0{\cdot}050\ \mu F$
$C_2 = 0{\cdot}020\ \mu F$
$C_3 = 0{\cdot}020\ \mu F$
$C_4 = 0{\cdot}010\ \mu F$
$C_5 = 0{\cdot}005\ \mu F$
$C_6 = 0{\cdot}002\ \mu F$
$C_7 = 0{\cdot}002\ \mu F$
$C_8 = 0{\cdot}001\ \mu F$

Fig. 10—Conductimetric method for carbon: measuring circuit

to the general method have been described by a number of workers in the U.S.A. and in the U.K.; it is fairly rapid and has for some years been established as the most accurate method available.

The large-sample method is of obvious value when the sample material is freely available and the equipment is in regular use. It shares with the conventional combustion method from which it was developed the disadvantage of being inconvenient for occasional use and it has the more serious limitation for certain work that the provision of sufficient sample would be often difficult and sometimes impossible.

A third method, less commonly used, involves conductimetric determination of the carbon dioxide produced in the combustion. This seemed to have advantages because (*a*) a conductivity cell and bridge would form a compact unit and would need little maintenance, (*b*) the sensitivity and precision of measurement would be related to the electrical techniques available and ought, therefore, to be high, (*c*) the carbon dioxide could be measured as and when it is evolved, without disconnection and removal of the absorption vessel. This is an important feature in the apparatus used for a great variety of samples, as there may often be some doubt as to whether all the carbon dioxide has been liberated within a specified time.

Apparatus

Apart from the usual oxygen-supply train and combustion furnace, several features of the equipment which has already been described[12] deserve special attention:

(i) The combustion-tube closure (Fig. 8) consists of an external brass tube made gas-tight with polythene and rubber sleeving holding a removable brass cover plate with inspection window and a right-angle gas inlet. The exit end of the combustion tube is closed by a rubber bung protected internally by a platinum baffle

(ii) The design of the conductivity cell (Fig. 9) has recently been improved, the platinum-foil electrodes described being replaced by thick silver rings heavily plated with platinum, which form a much more robust assembly. The conductivity cell is constructed of Perspex which permits maximum contact between the issuing gas and the 45 ml (approx.) of absorbing solution, the temperature of which is thermostatically controlled, e.g. by an external water bath

(iii) The measuring circuit (Fig. 10), which is a modified Wheatstone bridge of high sensitivity with a switch giving a 10:1 ratio of conductance readings.

Method of Operation

The usual precautions for low-blank operation are necessary, e.g. preheating of boats and flux (when required). Tin (for highly alloyed material) and lead (for iron-rich samples) are recommended, the latter being obtained with very low carbon content by melting at bright-red heat in a refractory crucible and pouring the molten globule (usually 0·7 g) on to the sample.

Combustion is carried out in the usual way at 1200–1250° C, iron-rich materials being dealt with satisfactorily at the lower temperature.

The oxygen flow is increased to 10 l./h immediately after inserting the boat and weighed sample into the mouth of the tube, the flow is reduced to 3 l./h after 5 min, and when the conductance reading becomes constant the boat is pushed into the hot zone.

The gas flow is increased as necessary to maintain a positive pressure during combustion (the exit tap may be closed temporarily if the increased flow is insufficient to prevent the absorbent being drawn back towards the combustion tube) and is again adjusted to 3 l./h when combustion is complete. When the conductance reading is again steady (25–30 min), a reading is taken; this must be corrected for a blank obtained under precisely similar conditions in the absence of a sample.

The percentage carbon is obtained by multiplying the change in conductance (microhms) by the cell factor (about 0·9 for sodium hydroxide, 0·5 for barium hydroxide) and dividing by $10^4 \times$ the sample weight.

Solutions for the conductivity cell need not be of exact strengths, 1 g/l. of barium hydroxide crystals or 1 g and 10 g/l. of sodium hydroxide being suitable dilutions. Appropriate ranges with these respective solutions are 0–1 mg, 1–10 mg, and 10 mg upwards of carbon. A few drops of foam-forming reagent are added to assist in ensuring complete absorption of carbon dioxide.

The factor of the absorbing solution may be obtained by combustion of known weights of calcium carbonate or sucrose in platinum boats, or by evolution of carbon dioxide from standard sodium carbonate solution added to sulphuric acid. It depends to some extent on the concentration of hydroxide in the solution, particularly with barium hydroxide for which a graphical factor correction is required.

With the sodium hydroxide absorbent it is possible to achieve reasonably reproducible results about 25 min after bringing the cell to equilibrium, a series of three or four determinations being made with each fresh charge of solution, but the great advantage of the procedure is its suitability for intermittent use while maintaining high reproducibility.

Acknowledgment

The authors wish to express their thanks to the manufacturers, The General Electric Co., Ltd. for their submission of the modifications to Fig. 9.

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An Investigation on the Function of Ingot Mould Dressings

By D. R. Thornton, B.Sc.

SYNOPSIS

This work was carried out to obtain information on the function of mould dressings, and the chemical and physical properties most necessary to reduce surface defects in ingots. Small-scale casting experiments were carried out in a double compartment mould with a transparent observation window so that the ingot surfaces obtained with various dressings could be related to the observed rise of metal in the mould. The physical and chemical properties of two types of mould dressings were determined, and a study made of the conditions at the mould surface.

From these experiments the factors affecting the occurrence of certain surface defects in ingots, and the advantages and disadvantages of certain dressings were found.

It is concluded that the volatile dressings offer most advantages, and of these the bituminous paints appear to be the best. Coal tar and charcoal based dressings are almost as effective in reducing surface defects, but are more difficult to use. 1254

ALTHOUGH it is generally accepted that, for many purposes, dressings on the surfaces of ingot moulds improve ingot quality, it has not yet been established in what manner this benefit is conferred. Previous mould dressing trials have been confined mainly to trial and error methods by direct comparison of ingot and billet quality obtained. Whilst this has been a guide to the usefulness and efficiency of the dressings, it has not given any information concerning their mode of action.

The types of ingot surface defect occurring most frequently on steel ingots are double skin, laps, cracks, and blowholes. Evidence from previous work on the subject indicates that the severity of ingot surface defects can often be related to the speed and temperature of teeming, and this has been represented graphically by Thomas* in Fig. 1. The units along the abscissæ and ordinates of such graphs vary with the type of steel, and for any steel there are ranges of values for teeming speed and teeming temperature which will give a minimum occurrence of surface defects on the ingot. These ranges are often very short.

Mould dressings offer a means by which the permissible ranges of teeming speed and temperature may be extended so that, although both vary during the pouring of a heat of steel, the quality of the ingots obtained is kept as high as possible.

The present series of experiments was designed to give some idea of the relative importance of various properties of mould dressings, to obtain information on their function, and the physical and chemical properties desirable to enable the dressings to fulfil this function satisfactorily.

The possible effects of mould dressings on the performance of the ingot moulds have not been studied.

Experimental Methods

An important point to be decided was the scale of operation. As the effects of many of the factors to be studied appeared to be independent of ingot size, it was decided initially to carry out small scale experiments.

The principal properties of the dressings involved were expected to be volatility, thermal conductivity (degree of insulation provided), and chemical nature, in particular whether the dressing produces reducing or oxidizing conditions.

A programme was arranged, therefore, in which dressings with various combinations of these properties were compared, so that their relative importance could be estimated. Apart from the type of dressing, other variables to be considered were mould temperature, teeming temperature, rate of teeming, type and condition of steel, and thickness of dressing applied. It was felt that this complex problem could best be approached by standardizing some of these variables and employing some method by which action occurring at the steel/mould interface would be visualized. A high-speed photographic technique was developed, and several series of experiments were carried out using this technique, initially with molten lead as reported below, and later with molten steel (p. 302).

The experiments may be summarized as:

(i) Study of surfaces of small blocks of low melting point metals cast with the use of various mould dressings
(ii) Photography of low melting point metals rising in moulds
(iii) Study of surfaces of small blocks of steel and photography of steel rising in the mould
(iv) Study of physical and chemical properties of mould dressing materials
(v) Study of conditions at the mould face during teeming
(vi) Photography of rising surface of steel in 10-cwt moulds.

EXPERIMENTS WITH LOW MELTING POINT METALS

Experimental Procedure

In the first experiments, molten lead was teemed as described below into a small, square mould,

* M. Thomas: Centre de Documentation Sidérurgie (Circulaire d'Information Téchniques) 1953, vol. 9, pp. 1421–1429.

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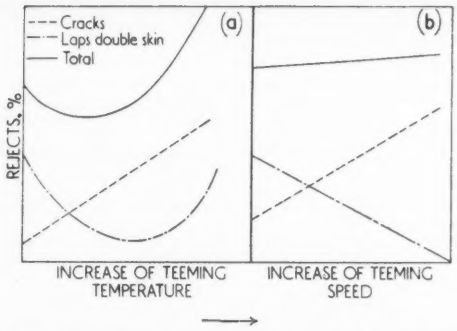


Fig. 1—Influence of teeming temperature and teeming speed on occurrence of surface defects

2 in. × 2 in. × 5 in. deep. One face of the mould was a clear silica or Pyrex glass plate, $\frac{1}{8}$ in. thick, through which the rising surface of the lead ingot was photographed. The other three faces were $\frac{1}{2}$-in. thick plates of planed cast iron.

The tundish used for teeming consisted of a length of silica tubing cemented into a hollowed brick to form a funnel. The tube was of such a length that, on resting the tundish on top of the mould in the position for pouring, the tube extended to about 1 in. from the bottom of the mould. Thus, on teeming the lead, turbulence was kept to a minimum, simulating bottom pouring. Tubes of different diameters were used to obtain variation in teeming speed.

The temperature of the lead was measured by means of a chromel/alumel thermocouple inserted into the crucible through the top of the furnace.

Slow-motion cine films were taken horizontally, normal to the silica plate, to show a magnified view of the meniscus and mould/metal interface, as the metal rose up one of the cast iron faces. A Paillard Bolex 16-mm cine camera was used at 64 frames/s, with 3 × $\frac{1}{4}$ in. extension tubes in conjunction with a 75-mm telephoto lens. The field of view obtained was about $1\frac{1}{4}$ in. × 1 in. and the speed was reduced by a factor of 4 when the film was projected.

Details of Tests and Observations

In the first series of casts, none of which were photographed, no dressings were used and the effect of variations in pouring temperature and teeming speed was observed on the formation of surface ripples on the ingot. When plotted in the form of a graph of pouring speed against temperature, as in Fig. 2, it can be seen that when no dressing is used, a line can be drawn, one side of which represents conditions leading to ripple formation, and the other side represents conditions under which ripples are not formed. Insufficient casts were made to verify whether or not the dressings used altered the position of this line. The results of the trials with dressings suggest that compounds which volatilize at a low temperature, e.g. oil and some waxes, tend to give deep, widely spaced ripples, whilst the bituminous paint,* Apiezon wax, and refractory washes, i.e. materials which volatilize little or not at all at the temperatures involved and which provide thermal insulation, tend to give very fine ripples or inhibit ripple formation altogether.

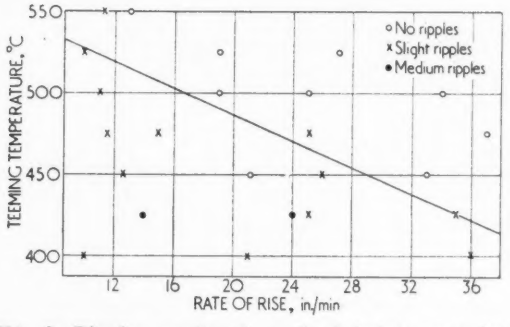


Fig. 2—Ripples produced on lead ingots cast into non-dressed mould

Table I
OBSERVATIONS ON FILMS USING MOLTEN LEAD

Dressing	Temp. of Lead	Rate of Rise	Observations
Lard oil Graphite in oil	400° C 400° C	30 in./min	Deep ripples formed, chilling rate apparently increased—probably due to low teeming temperature
None	450° C	5 in./min to 18 in./min	Smooth even rise, occasional lap with slow teeming
Bituminous Paint No. 1	450° C	10 in./min to 12 in./min	Smooth even rise with slow teeming, occasional deep ripple or over lap. Dressing appears to melt and form a liquid cushion between metal and mould, thus reducing chill rate
Water	450° C	10 in./min	Deep ripples formed, chilling rate apparently increased—possibly due to high latent heat of water
Paraffin wax Thick (3–4 mm)	450° C	5 in./min	Wax is slow to melt, appears to increase the chilling rate, thus producing deep ripples or laps
Paraffin wax Thin (0·5 mm)	450° C	10 in./min	Smoother rise but effect similar to that for thick wax only less pronounced. Each time the metal meniscus appears to fold against the wall forming a ripple, a puff of wax vapour is emitted
Stearic acid	450° C	20 in./min	Fast rise giving many fine ripples, general rise is smooth. Effect seems to be similar to paraffin wax
None (Degreased mould)	500° C	14 in./min	Smooth rise
None	500° C	60 in./min	Fast jerky rise due to uneven teeming
Shellac	500° C	30 in./min	Fast jerky rise due to uneven teeming. There may be a variation in contact angle or perhaps the surge merely gives that effect
Sairset refractory (3-4 mm)	500° C	25 in./min	Smooth even rise but metal can still be seen to fold back forming ripples

* The bituminous paints tested are proprietary mould dressings consisting of a bitumen base thinned with solvent.

Table II

TIME FOR METAL TO STICK TO PLATE

Results for various dressings using different materials

	Thickness of Dressing, mm	No Dressing, s	Stearic Acid, s	Bituminous Paint No. 1, s	Alumina Cement, s	Charcoal* Based Dressing, s
Lead (450° C)	0·5		1	3	>10	0
	1·0	0	2	5	>10	1
	2·0		4	6	>10	>10
	4·0		4	8	>10	>10
Aluminium (750° C)	1	>10	>10	>10	>10	>10
Bronze (1100° C)	2	0	2	>15	6	...

* A proprietary dressing, principally charcoal and borax, made to a paste with water

In the second series of casts, various dressings were used and the teeming was photographed with the cine camera. Study of these films indicates that the mechanism of ripple formation is the rapid chilling of the metal surface as it comes into contact with the mould wall. Hence, the lower the teeming speed or metal temperature, the more likely is ripple formation. In some cases, when the teeming speed and temperature were low, initial freezing of the metal surface extended over the meniscus giving rise to surface laps. Observations on the films of the second series are given in Table I.

In the third series of casts, molten metal was poured from a height of 3 in. on to coated cast iron plates at room temperature, inclined at an angle of 45° to the horizontal, to compare the efficiency of various dressings in preventing the sticking of 'splash' and 'surge.' Tests were carried out with lead, aluminium, and bronze, and the results obtained are given in Table II. The time from the start of pouring until the metal first began to stick to the plate was taken as a measure of the efficiency of the dressing.

These experiments indicated that the nature of the dressing can exert a considerable effect on the occurrence of sticking. The bituminous paint was more effective in the prevention of splash sticking with bronze than with lead, probably because of the higher temperature involved, resulting in a greater amount of gas evolution. The dressing properties mainly responsible for reducing sticking are not known but the following may be most important: (*a*) propulsive effect of gas evolution; (*b*) thermal capacity of the dressing; (*c*) latent heat of vaporization of the dressing; (*d*) surface tension metal/mould and contact angle; (*e*) exothermic effect.

EXPERIMENTS WITH STEEL

Experimental Procedure

Moulds and Teeming Technique—The first type of mould used (Fig. 3*a*) was 6 in. long, 2½ in. wide, and 5 in. high. A brick partition dividing the mould into two roughly equal sections was placed parallel to the Pyrex glass plate. The brick extended to about 1½ in. from the base. The steel was teemed into the back section of the mould and the rising meniscus in the front section was photographed through the glass, with the brick partition as a background. The effect of this was to cut out any glare from the teeming stream and to simulate bottom pouring in the front section of the mould, thus considerably reducing turbulence.

In order to compare directly the effect of two dressings whilst keeping other conditions as constant as

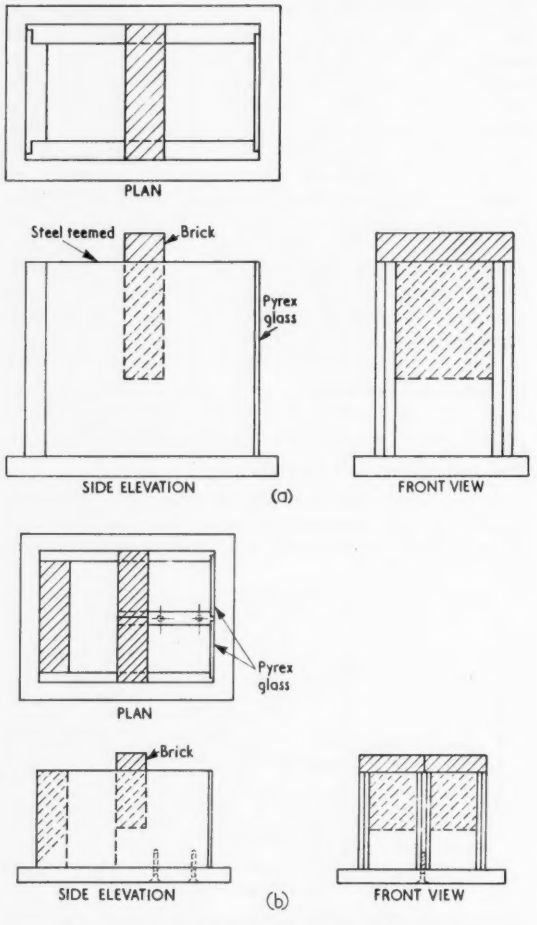


Fig. 3—(*a*) Single mould (*b*) Double mould

Table III
DRESSINGS USED

Dressing	Characteristics	Thicknesses used, mm
Bituminous Paint No. 1	Insulating; Reducing; Volatile	0·05; 0·1–0·5; 1·0
Alumina cement	Insulating	1·0–2·0
Charcoal based dressing	Insulating; Reducing; Volatile	2·0–3·0
Stearic acid*	Reducing; Volatile	1·0
Soot (from burning tar)	Reducing	0·5–1·0

* Stearic acid tended to melt and run off before teeming

possible, a double mould, shown in Fig. 3*b*, was used. This mould was 6 in. wide, 9 in. long, and 5 in. high. A cast iron partition fixed to the base plate divided the front section of the mould into two compartments, thus enabling the rise of metal against two faces to be photographed simultaneously.

Immediately after teeming, the sides of the mould were removed from the outer faces of the ingots; the inner faces were allowed to cool in contact with the cast iron partition. This gave a rough indication of the effect of rapid and slow stripping respectively

Table IV
EXAMINATION OF STEEL INGOT SURFACES (SERIES 1)

Cast	Dressing	Surface	Laps
A	No dressing	Fairly good to moderate Shallow ripples Sunken areas	Slight
	Bituminous Paint No. 1	Fairly good Shallow ripples	None
B	Reeking	Moderate Ripples Splash	Slight
	Bituminous Paint No. 1	Moderate Deeper ripples Sunken areas	Slight
C	Reeking	Fairly good Ripples	Bad
	Bituminous Paint No. 1	Good Shallow ripples	None
D	Bituminous Paint No. 1	Good Very shallow ripples	None
	Alumina cement	Fairly good Ripples Splash	Medium
E	Bituminous Paint No. 1	Fairly good Shallow ripples	None
	Alumina cement	Moderate Ripples Sunken areas	Medium
F	Alumina cement	Moderate to rough Ripples Splash	Medium
	Bituminous Paint No. 1	Fairly good Shallow ripples	None
G	No dressing	Fairly good Shallow ripples	None

Table V
EXAMINATION OF STEEL INGOT SURFACES (SERIES 2)

Cast No.	Dressing	Surface	Laps
1	Bituminous Paint No. 1 (Medium)	Deep ripples with subsequent metal flow into hollows (double skin)	No
	Bituminous Paint No. 1 (Thin)	Moderate ripples and sunken area	No
2	Stearic acid	Moderate ripples, some slag entrapped in surface	No
	Bituminous Paint No. 1 (Thin)	Moderate ripples, some slag entrapped in lower half with slight blowholes	No
3	Bituminous Paint No. 1 (Medium)	Moderate ripples, subsequent metal flow into hollows	No
	Bituminous Paint No. 1 (Thin)	Fine to moderate ripples	No
4	Bituminous Paint No. 1 (Thick)	Moderate ripples, some deep cavities as steel over-ran dressing, subsequent metal flow into hollows	No
	Bituminous Paint No. 1 (Thin)	Fine to moderate ripples, surface slag entrapped in skin, some cavities	No
5	Bituminous Paint No. 1 (Thin)	Fine ripples, slight slag entrapment on surface	No
	Charcoal based dressing (Thick)	Good surface except for some blowholes, extremely shallow ripples	No
6 Nitrogen Atmosphere	No dressing	Fine ripples, slight slag entrapment	Yes
	Alumina cement	Bad surface cavities, slag entrapment	Yes
7 Nitrogen Atmosphere	No dressing	Fine ripples, good surface	Yes
	Soot (Tar reeked)	Very fine ripples, smooth surface, some adhered splash	Yes

on the condition of the ingot face. For teeming, a gas-heated tundish, consisting basically of a plumbago crucible with a $\frac{5}{16}$-in. zircon nozzle, was constructed. The crucible was preheated to a temperature of 1420–1460° C and was filled with steel taken from a 10-cwt ladle used on the arc furnace. Temperatures taken with a Pt/Pt–Rh thermocouple in the crucible during teeming were within the range 1470–1500° C and there was thus a small but variable superheat in all the casts. A silicon-killed steel of 0·4–0·6% C was used.

Photography—The photographic technique was similar to that used for the lead ingots. It was thought, however, that more useful results might be obtained if it were possible to slow down the motion in the projected film by a higher factor than 4. For this purpose a Kodak III high-speed camera run at 750–1000 frames/s was used, thus slowing down the motion on projection by a factor of up to 62·5. The 120 mm lens gave a slightly larger field area than was obtained with the Bolex camera, i.e. $1\frac{1}{2}$ in. × $1\frac{1}{4}$ in., and the camera was set up so that the rise of steel against both faces of the centre partition of the mould could be photographed simultaneously at a height of 2–3 in. from the base of the mould. Films taken at 64 frames/s with the Bolex camera were also obtained of several of the casts.

Ingot Testing—All the resulting ingots were examined for surface quality, and micro and macro sections were cut from regions adjacent to the

Table VI

OBSERVATIONS ON FILMS (SERIES 1 AND 2)

Cast	Dressings		Temp. of steel in tundish	Rate of rise	Observations	
	LHS	RHS			LHS	RHS
Series 1 Cast A	No dressing		1480° C	30 in./min	Metal rises smoothly, oxide coating continuous over meniscus, moisture or other volatile matter present at 2/3 height of field, causing turbulent motion of steel pushing back and breaking up the oxide film	
Cast C	Soot (tar reeked)	Bit. Paint No. 1, 0·1–0·2 mm	1475° C	18 in./min	Oxide coating continuous over meniscus, steel appears to rise in stages, breaking through the upper surface and flowing over the existing meniscus, rising ¼ in. or ½ in. then pausing before the whole cycle is repeated. This causes ripples or teeming laps	Metal rises smoothly, oxide coating is pushed back from meniscus by a surface flow of steel away from the mould/metal interface
Cast F	Alumina cement	Bit. Paint No. 1, 0·1–0·2 mm	1490° C	25 in./min	Oxide coating continuous over meniscus, steel appears to rise intermittently, breakthrough appears to occur when sufficient pressure has built up in the metal head	Although the meniscus is partly obscured by a background glare, surface flow normal to the interface is evident, keeping back the oxide coating from the meniscus
Series 2 Cast 2	Stearic acid (melted, thus very little on walls)	Bit. Paint No. 1, 0·05 mm	...	15 in./min	A tendency for the surface metal to flow away from the interface is evident but is not fully effective in pushing back the oxide coating, metal observed to break through the base of the ripples when the pressure built up (¼ in. below surface), ripple formation similar to that of Bituminous Paint No. 1 coated moulds	This very thin layer of Bituminous Paint No. 1 was not effective in keeping back the oxide film; no evidence of surface flow was visible, metal rise was smooth in both sections
Cast 3	Bit. Paint No. 1, 0·5 mm	Bit. Paint No. 1, 0·05 mm	...	12·5 in./min	Splashes from the dressing almost completely obscured the glass 'window.' Vigorous reaction at the interface could be seen; dressing burned with evolution of gases, steel and unburnt dressing were pushed or thrown clear of the interface	Very slightly more effective in keeping the oxide film clear of the interface than in cast 2, metal rise smooth
Cast 4	Bit. Paint No. 1, 1 mm	Bit. Paint No. 1, 0·05 mm	1475° C	12·5 in./min	Glass window almost completely obscured by soot from the burning dressing, which was only burnt away about ¼ in. below the meniscus	Dressing not fully effective in keeping the oxide film clear of the interface; presence of burning gases and fumes appears to prevent a complete oxide film forming
Cast 5	Bit. Paint No. 1, 0·05 mm	Charcoal based dressing 2–3 mm	1470° C	25 in./min	Dressing was not very effective in keeping the oxide film clear of the interface	Almost completely obscured by soot deposited by the burning dressing; vigorous reaction seen at the interface, dressing and steel being thrown clear of the mould/metal interface. Metal overruns the dressing, metal rise smooth
Cast 6	No dressing, nitrogen blown through mould from top	Alumina cement	1470° C	22 in./min	Metal appears to rise more smoothly and evenly than in the RHS. Very little oxide on surface of steel	Deep ripples can be observed in formation as meniscus does not rise steadily but is continually being folded back against the mould face as the pressure in the metal head increases; nitrogen blast was stronger in the RHS of the mould and possibly caused greater cooling
Cast 7	No dressing, nitrogen blown through mould from top	Soot (tar reeked)	1470° C	21 in./min	Metal rise reasonably smooth but in lower part of field metal freezes before coming into contact with mould wall even as it is being pushed towards the wall by the pressure behind. As steel at the top folds against the wall, the rest falls into line subsequently due to increased pressure from behind	Rise of metal reasonably smooth although pauses occur when metal from alongside flows over the existing meniscus to give a teeming lap; at times a surface metal flow normal to the mould wall near the interface can be seen but not as effectively as with bituminous paint No. 1; film at 64 frames/s also shows this surface flow, very little oxide was present on the surface

ingot surface to show subsurface structure. These sections were mostly taken from the position in the ingot where the steel rise was photographed, in order to correlate the surface and structure with the observed reaction at the interface.

Details of Tests and Results

The dressings used in the experiments and the characteristics they were expected to show are given in Table III.

Coal tar dressings were not included in the first two series of experiments because preliminary tests had indicated that they were very similar in action to bituminous paints.

Experiments on 25-lb Mould

The results of the examination of the steel ingot surfaces obtained in the first and second series of tests are given in Tables IV and V.

It was observed from this examination that two distinct types of ripple were obtained on the ingot surfaces:

(i) With volatile dressings the ripples tended to be undulating in nature

(ii) With non-dressed moulds and moulds coated with non-volatile dressings the ripples were shallower, less widely spaced, and resembled tiny overlaps.

The charcoal based dressing gave an almost ripple free furface.

The charcoal based dressing and bituminous paint No. 1 gave less lapping than the non-volatile dressings. The thickness of bituminous paint No. 1 seemed to have little effect on ripple or lap formation, but thick layers gave rise to ' bleeding ' and over-running of the dressing by the steel. Alumina cement, in spite of its heat-insulating properties, tended to increase the incidence of surface laps.

The relationship between the rate of metal rise in the mould and the ripple frequency on the ingots, for the casts in the first and second series, is plotted in Fig. 4. From this it can be seen that although no curve can be constructed which will relate the frequency of the ripples with the rate of rise for all types of dressing, a curve can be drawn for the volatile dressings alone. The points obtained with no dressing and with non-volatile dressings lie above this curve.

In the third series of tests, the oxidation of the steel surface was reduced considerably by casting the ingots in an atmosphere of oxygen-free nitrogen. Unfortunately, no precautions were taken to ensure that this nitrogen was dry and some oxidation of the steel in the moulds did occur. In general, it was noted that the incidence of lapping with non-volatile dressings, series 3, was less than in the earlier tests, but definite conclusions cannot be drawn since the teeming speeds varied considerably, and the nitrogen tended to chill the steel in the moulds.

The general tendency, however, appears to be that reduced oxidation of the steel meniscus in the mould decreases the incidence of surface lapping. There was one exception to this among the dressings tested. With alumina cement coated moulds, a material of high luminosity could be observed forming at the steel/mould interface during teeming. This spread over the meniscus and gave rise to a jerky and uneven metal rise with consequent formation of laps.

Tables VI and VII give observations from the cine films of series 1, 2, and 3. These observations, in conjunction with those of the surfaces of the ingots obtained (Tables IV and V), indicate that the presence of oxide or slag on the steel meniscus is largely responsible for the formation of surface laps.

This is illustrated clearly by series 1, cast C (*see* Tables IV and VI). The tar reeked section of the mould gave rise to a badly lapped ingot—the bituminous paint No. 1 coated section gave an ingot free from laps. Photographic observations of the rise of metal in the moulds showed that the meniscus in the tar reeked section was covered with a layer of oxide throughout the teeming, leading to overlapping of the metal, whilst the meniscus in the bituminous paint No. 1 coated section was practically free of oxide, giving a smooth metal rise.

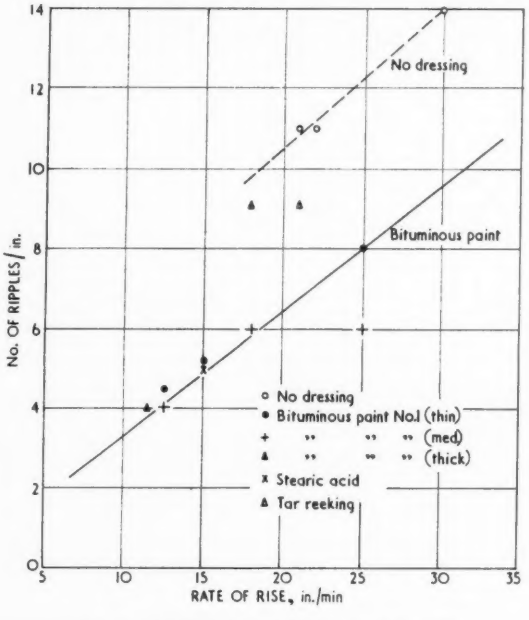


Fig. 4—Ripples produced on steel ingots

Experiments on Top-poured 10-cwt Mould

In the fourth series of tests, casts were made in a 10-cwt mould, in the wall of which a glazed silica plate had been fitted. These casts were photographed in order to ascertain whether conditions during teeming were comparable between 25-lb and 10-cwt ingots.

Table VIII gives the results of 5 casts. The films showed that little more turbulence occurred than in the small moulds. The ingot surfaces obtained indicated that the charcoal based dressing, bituminous paint No. 1 and tar dressings were the most effective in reducing the sticking of splash.

Table VII

OBSERVATIONS ON FILMS

Oxygen-free nitrogen passed through moulds (Series 3)

Cast	Dressings		Temp. of steel in tundish	Rate of rise	Observations	
	LHS	RHS			LHS	RHS
1	Alumina cement	No dressing	...	25 in./min	A blanket of oxide or slag-like material formed at interface spread over the meniscus, resulting in a jerky rise with lap formation	Steel appeared to be cold, surface was probably chilled by stream of nitrogen; steel surface was almost free of oxide, and after an initial lap the rise was smooth
2	Charcoal + 15% bentonite	Charcoal based dressing (proprietary)	1465° C	12 in./min	Surface covered with layer of oxide giving jerky rise with lap formation; gas evolution occurred below the steel/mould interface after each overlapping of the steel	Violent turbulence at surface, later a jerky rise was obtained with no apparent gas evolution
3	Charcoal based dressing	Coal tar pitch based dressing	1485° C	40 in./min	Violent turbulence	Tendency for the over-running and entrapment of dressing near base of mould; fumes were emitted which filled the mould; steel meniscus was clear of oxide; smooth rise
4	Graphite wash	Tar reeked (soot)	...	60 in./min	Continuous surface flow over meniscus normal to mould wall, high contact angle, smooth fast rise with some break through of metal 1–1½ in. below surface	Smooth rise; was some difficulty in seeing the condition of the meniscus as the film was slightly out of focus
5	Tar reeked (soot)	Alumina cement	1460° C	300 in./min	No satisfactory film was obtained as the rise was too rapid; there appeared to be a fast, smooth rolling of the steel up the mould wall with intermittent pauses	As LHS
6	Graphite wash	Charcoal + 15% bentonite	1480° C	8 in./min	Steel rose only through ¼ of the camera field, very slowly; meniscus was covered with oxide and the steel rise was jerky with lap formation; contact angle appeared to vary	Meniscus was free of oxide, and steel rise was smooth with no apparent gas evolution

DETERMINATION OF PHYSICAL AND CHEMICAL PROPERTIES OF DRESSINGS

The types of mould dressing examined were: (*a*) Coal tar dressing the raw materials for which are pitch, obtained from the distillation of coal, and coal tar naphtha, which is mainly unsaturated and aromatic; (*b*) Petroleum based mould dressings, which consist of bitumen, obtained from the distillation of crude oil, with heavy fuel oil or mineral spirits, the latter being dominantly a mixture of saturated

hydrocarbons with some unsaturated and aromatic hydrocarbons. In some cases the aliphatic bitumen is replaced by cyclic residues recovered during the processing of tar and benzene. Benzene can be employed as the solvent.

The results of tests on factors affecting the application of the dressings to the mould and the function of the dressing during teeming are given in Tables IX and X.

Distillation curves are shown in Fig. 5. Both curves start at 150°C, but that for the bituminous paint rises initially much more steeply, indicating that this material contains a greater proportion of lower boiling point volatiles than the coal-tar dressing.

About half the volatile content of bituminous paint No. 1 is evolved as the paint dries on the mould wall (when the mould is dried at 100°C). The curve for the coal tar pitch dressing rises at an almost constant rate and only 40% of the volatile constituents are lost in drying.

Although slightly more volatile constituents are driven off the coal tar dressing during teeming, they are evolved at a temperature about 50°C higher than for the bituminous paint.

To obtain information on the degree of thermal insulation provided by the various mould dressings, a series of experiments was carried out involving the dipping of a 2 in. × 3 in. × 6 in. cast iron machined block into the electric furnace bath. One of the two opposite faces of the block was coated with a dressing, the other face being uncoated, and the block was dipped into 0·35–0·45-C Si-killed steel at 1500° ± 10° C for 5 s. The mean thickness of solidified steel on the faces of the block was determined. The figures obtained as a mean of several dip tests for each dressing were taken as an indication of the ' thermal barrier ' effect of the dressing. These are:

Bit. Paint No. 1	Alumina cement	Coal-tar pitch with volatile solvent	Char-coal	Paper	Pitch suspension
0·86	0·53	0·86	0·88	0·81	0·90

These figures are given as ratios, the thickness for an uncoated face being taken as unity. They indicate that the initial thermal insulation provided by bituminous paint No. 1, the coal tar pitch based dressing, charcoal, paper, and pitch suspension, are not substantially different. As might be expected, alumina cement offers a greater degree of insulation.

Table VIII

OBSERVATIONS ON TRIALS IN 10-CWT MOULDS

Cast	Dressing	Remarks
1	Charcoal based	Good smooth surface, no ripples; no splash, no laps; cine film observations as with small ingots
2	Refractory paste	Fairly good surface, no ripples; slight splash, slight tendency to lap formation; cine film shows rather jerky rise; no noticeable extra turbulence over rise in small mould
3	Bituminous paint No. 1	Fairly good surface, undulating ripples; slight splash, no laps; cine film observations as for small moulds; meniscus clear of oxide; smooth rise
4	No dressing	Fairly good surface except for extensive splashes; tendency for lap formation; cine film observations as for small mould
5	Tar	Fairly good surface, slight splash; no laps; cine film observations, smooth rise with meniscus clear of oxide

In all Casts: Initial mould temperature 50–60° C; Rate of rise 45in./min; Filmed at 64 frame/s.

Table IX

FACTORS AFFECTING APPLICATION

Test	Bit. Paint No. 2	Bit. Paint No. 1	Coal tar pitch with volatile solvent
Viscosity 70° F	377 s	2350	...
Redwood No. 1 90° F	212 s	1330	...
100° F	170 s	1080	2000
Sp. gr.	0·901	0·925	1·156
Drying time (relative)	1	1	9
Flash pt., °C	45	45	51·5
Loss of solvent during drying, %	42	38	28
Water content, %	nil	nil	0·25

STUDY OF THE CONDITIONS AT THE MOULD SURFACE

Theoretical Considerations

The effect of the dressings, in particular the volatile dressings, depends partly upon the amount of heat absorbed ahead of the rising steel surface. If the dressing absorbs sufficient heat by radiation, by con-

Table X

FACTORS AFFECTING FUNCTION

	Bit. Paint No. 2	Bit. Paint No. 1	Coal tar pitch with volatile solvent
Coating Surface	Glossy brown/ black	Glossy brown/ black	Glossy black
Volatiles – % of evolved initial wt.	44	43·9	47
Coking – „ Residue	14	18·1	24
Ash – „ Content	0·07	0·07	0·01

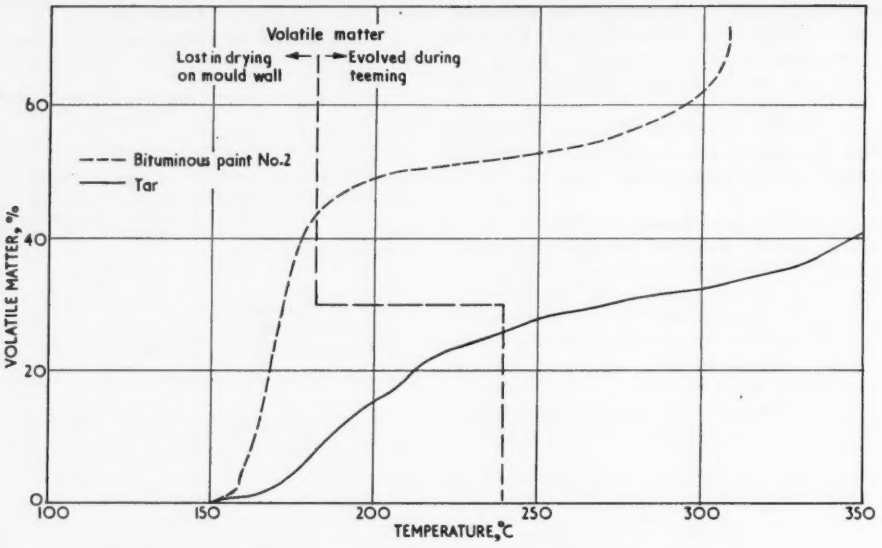


Fig. 5—Distillation curves of tar, and bituminous paint No. 2

duction through the mould wall, and by convection, it will vaporize above the steel meniscus and help to determine the amount of surface oxidation of the steel which takes place. It may give a gas 'cushion' insulating the steel from the mould wall, or it may merely melt and give a liquid cushion. On the other hand, nothing may happen until the steel touches the dressing.

It can be shown mathematically, that for volatile dressings the heat absorbed by the thin layer of dressing would be sufficient to cause vaporization, providing that the heat were not conducted away too rapidly through the mould wall. The argument is outlined below.

Heat Transfer by Conduction—A certain amount of heat is conducted through the mould wall ahead of the steel meniscus.

Using an expression which is available for estimating the temperature of the mould wall at any point, it was calculated that a position level with the top of the meniscus (see point *A* in Fig. 18) would, under equilibrium conditions, be approximately 20° C hotter than the original mould, and a position very close to the bottom of the meniscus (see point *B* in Fig. 18) would be 90° C hotter, due to heat conduction through the mould wall.

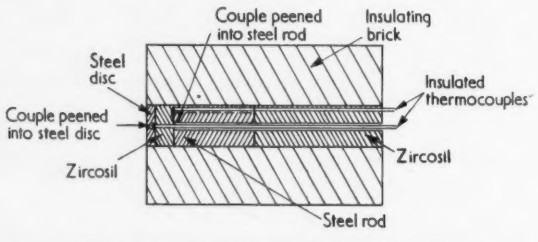


Fig. 6—Rapid-response heat flow-meter

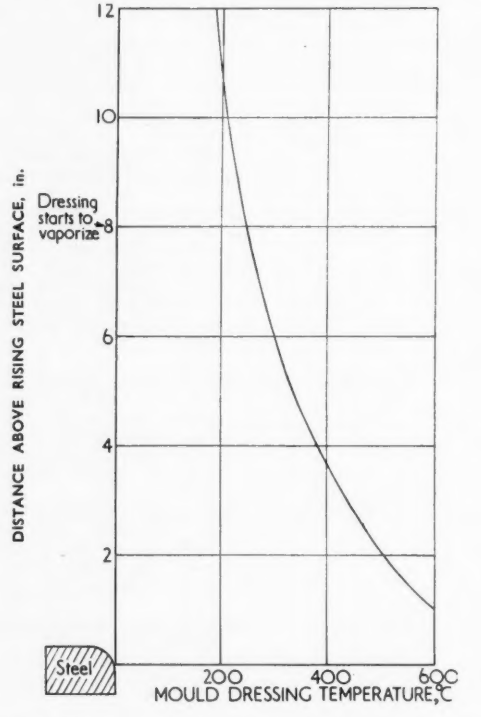


Fig. 7—Temperature rise of tar-based dressing applied 1-2 mm thick; rate of rise 0·88 in./s

Heat Transfer by Radiation—When the rising steel approaches any point on the mould wall, heat is absorbed at the point by radiation from the steel surface at a rate given by:—

$$Q = \frac{\sigma T^4}{2} \text{ cal/cm}^2\text{/s}$$

During the period when the point is at a level between those of the top and bottom of the meniscus the rate of heat absorption increases to a value approaching σT^4. Thus the heat transferred to the mould wall is about 7·0 cal/cm²/s rising to a maximum of 14 cal/cm²/s immediately above the steel/mould interface.

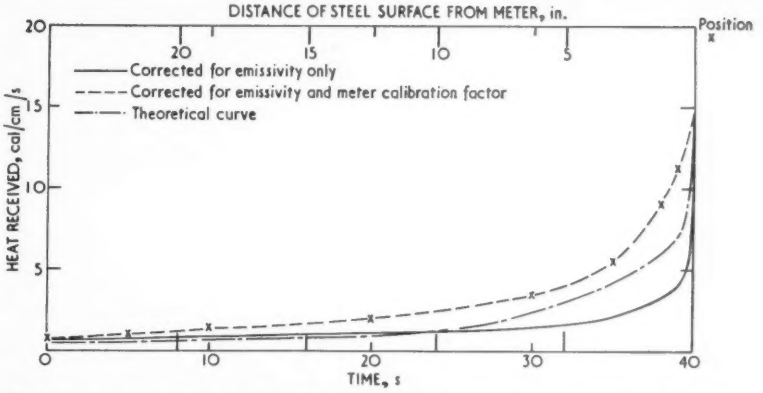


Fig. 8—Heat received at position X on mould wall by radiation from steel surface; 10-cwt mould, height of position × 25 in.

[*Note:* The ordinate scale should be "cal/cm²/s"]

Considering a point on a mould wall 1 in. above the interface, with the steel rising 20 in./min, then that point will receive about 30 cal per unit area before the steel reaches it. This should be sufficient to vaporize a thin layer of a volatile dressing provided that the heat is not transmitted too quickly through the mould wall.

In order to check this, experiments were carried out to determine the temperature rise of the mould wall ahead of the molten steel during teeming.

Measurement of Temperature Rise of the Mould Wall

A 10-cwt mould was used with a chromel/alumel couple immersed in a tar-based dressing painted on the mould wall. The dressing was applied to a thickness of 1–2 mm, to secure the couple in position and ensure that it was insulated from the wall of the mould. A rapid-response heat flow meter was also constructed (Fig. 6), consisting of a mild-steel disc, into which a chromel/alumel thermocouple had been peened, insulated by a 7-mm thick layer of Zirconal from a steel rod of the same diameter. This was inserted in a cylindrical insulating brick case 2 in. dia. The meter was calibrated by means of a black-body furnace before use.

The temperature rise was measured by means of a 2-channel quick-response recorder in conjunction with an amplifier, and the results obtained are plotted in Figs. 7 and 8.

Figure 7 shows that the rise in temperature of the mould dressing above the surface of the steel was considerable, and vaporization of dressing ahead of the metal would certainly have occurred. The position of this curve is dependent upon several factors:

(i) Rate of rise of steel in mould
(ii) Temperature of teeming
(iii) Dimensions of mould
(iv) Thickness and physical properties of dressing.

Figure 8 was constructed to show the heat radiation to the mould wall from the steel. The theoretical curve has been corrected for the dimensions of the mould and the experimental curve agrees fairly well with this.

The amount of heat radiated per second to the mould wall is independent of the rate of teeming, but the total amount of heat radiated is, of course, inversely proportional to the rate of rise of the steel. A curve showing the amount of heat received by the mould wall could be constructed for any type of mould, and in theory could be used to indicate the best type of dressing and the thickness required according to the volatility and latent heat of the dressings available.

FACTORS AFFECTING THE SURFACE DEFECTS FOUND

Whilst it is realized that an exhaustive series of trials would be necessary to cover every possible variable appertaining to the study of mould dressings, it is felt that useful information has been obtained from the few experiments so far carried out. This is discussed below.

Splash and Surge—Evidence obtained from casts into 10-cwt moulds showed that of the dressings tested, bituminous paint No. 1 and the charcoal based coatings were most effective in preventing sticking of steel splashed on the mould walls. The sloping plate test described above (p. 302) indicated that with a low melting point metal (lead), a coating of refractory wash would be more effective than a volatile dressing, but with higher melting point metals the volatile dressings would be more effective than the refractory wash. The 10-cwt ingots tended to confirm this.

It is evident that heat insulation properties alone in a dressing are not sufficient to prevent the sticking of splash and that the volatility of the dressing is an important factor. The action involved is not known with certainty but it is probably a combination of the following factors:

(i) Carburization of the steel splashes by the reducing gases evolved from the dressing, lowering the melting point of the steel
(ii) Propulsive effect of gas evolution
(iii) Prevention of direct contact between steel and mould
(iv) Exothermic effect of burning dressing.

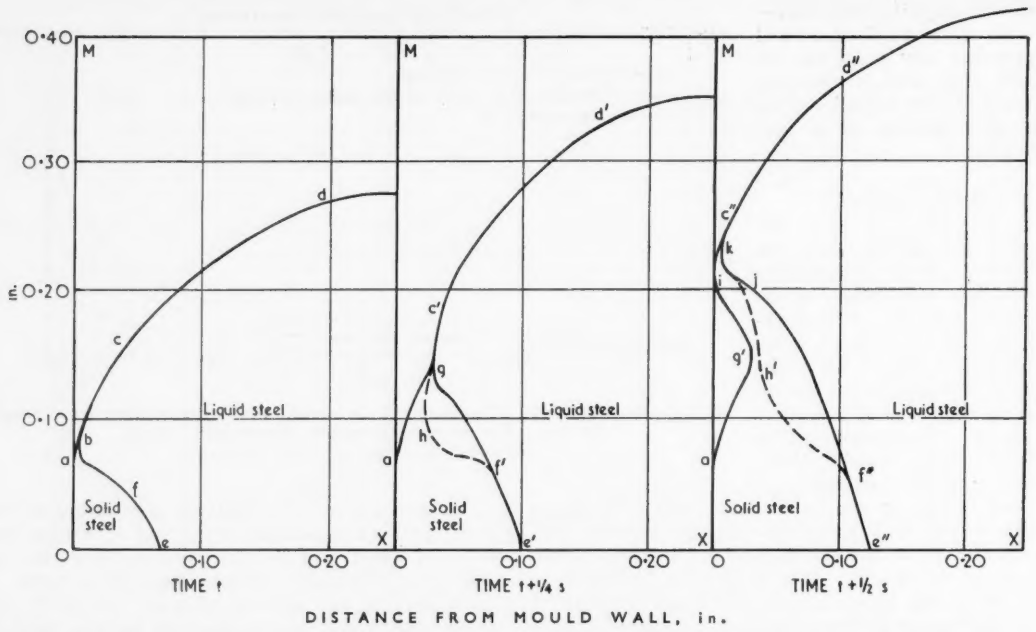


Fig. 9—Rise of steel meniscus up the mould at various time intervals; mould coated with bituminous paint No. 1; rate of rise 18 in./min

Double Skin Effect—Several of the small block ingots from moulds coated with thick layers of bituminous paint No. 1 showed ' bleeding ' that had occurred in the hollows of the surface ripples. In one or two of the ingots this led to a type of double skin away from the ingot bottoms and a little surface blowing.

As shown in Fig. 9, the thickness of solid skin in the hollow of the surface ripples is probably initially very small. Ferrostatic pressure causes a ' flattening ' of this hollow, and in some cases a breakthrough of molten metal, giving a double skin, particularly when the teeming temperature is high, or with the use of a thick layer of dressing.

Ripples—The formation of surface ripples on ingots may be affected by the rate of heat extraction by the mould and the properties of the liquid steel, particularly the surface tension. The shape and height of the meniscus, under equilibrium conditions, are dependent upon the surface tension of the steel and the steel/mould contact angle.

Taking the surface tension of the steel as roughly 1200 dyn/cm², then the height r (Fig. 10) of the meniscus can be shown to be of the order 0·2–0·25 in. The contact angle will vary according to the degree of molecular attraction between the steel and the mould wall. It is generally of the order 140–150°, but if a dressing is used which insulates the steel completely from the wall, then the contact angle may be increased to values approaching 180°.

The presence and distribution of surface ripples is dependent to a great extent on the rate of chilling of the steel meniscus as it rises up the mould wall and the degree of oxidation of the steel surface. The chilling appears to depend mostly on the degree of volatility of the dressing. The greater the amount of gas evolved from the dressing, the more widely spaced are the ripples. This may be due to the increased gas pressure, $P1$ on the meniscus near the interface holding back the steel until sufficient ferrostatic pressure has been built up, $P2$, so that a periodic folding back of the meniscus against the mould wall ensues.

Thus the following factors are involved:

(i) Initial mould temperature
(ii) Degree of superheat of steel

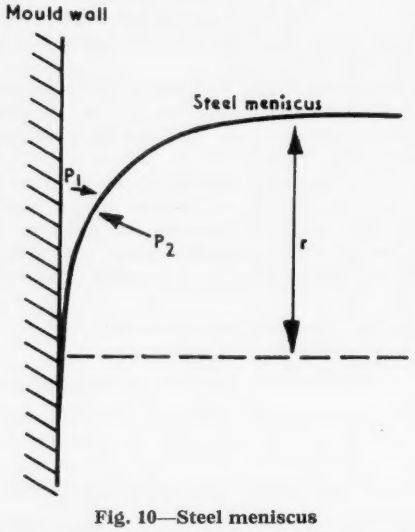


Fig. 10—Steel meniscus

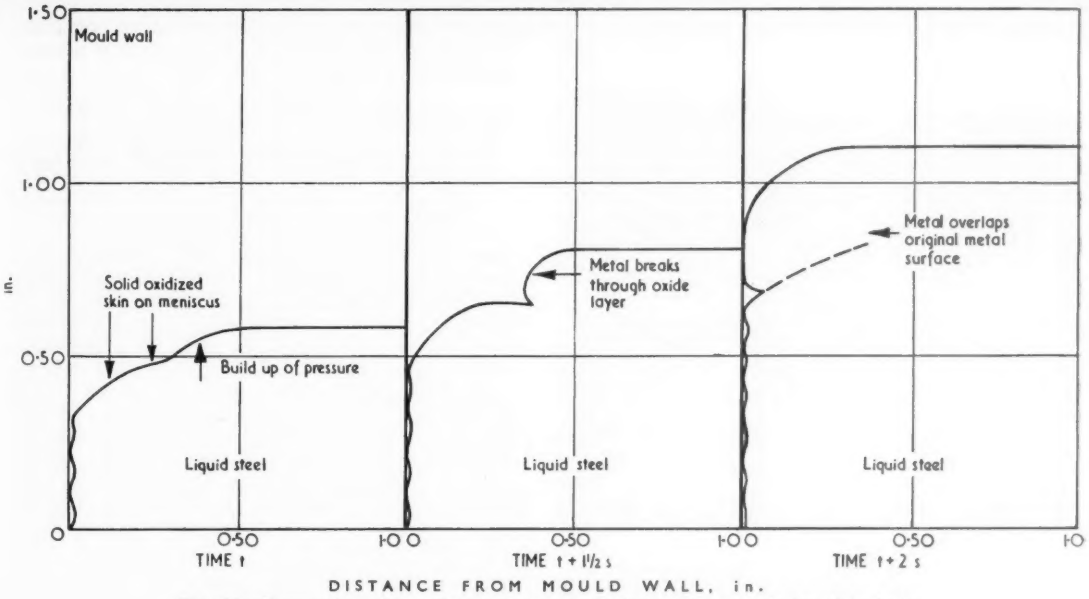


Fig. 11—Formation of laps in ingot, tar-reeked mould; rate of rise 18 in./min

(iii) Teeming rate
(iv) Oxidation of steel surface
(v) Volatility of dressing.

The ripples are generally in the form of shallow, narrowly spaced overlaps with an uncoated mould or with a mould coated with a non-volatile dressing. When a volatile dressing is used, the ripples are more widely spaced and undulating in nature.

In general, the higher the values of each of the first three factors, the greater the number of ripples per unit distance and the less severe is the defect.

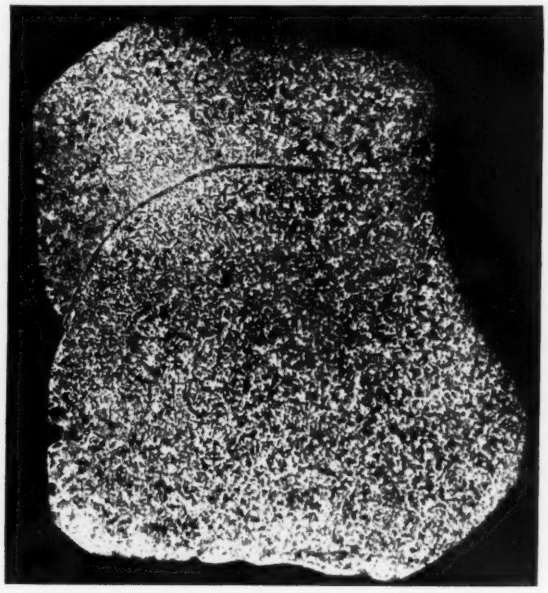


Fig. 12—Lap in tar reeked ingot × 5½

It was observed from the cine films that when ripples of the narrow over-lapping type are formed, the steel/mould contact angle tends to be small, (120–140°), and the meniscus is short and flat, and usually covered with an oxide layer. As a result of this, freezing does not generally extend far above the interface and the resulting ripples are narrow.

With a volatile dressing, the contact angle tends to be high (140–160°), and the meniscus is tall and mainly free from oxide. Under these conditions the gas pressure between the mould and the meniscus tends to hold back the meniscus and, in addition, freezing may extend a little further over.

It is probable that the delay in the chilling of the ingot surface, even with a volatile dressing, is very small, hence the rate of chill has been calculated as though the steel and mould were in contact. As there is no mould/steel contact at the interface, the meniscus tends to build up until, when the internal pressure becomes too great, the meniscus bends back to the mould wall at the limit of the frozen surface layer, as shown in Fig. 9. With a relatively low rate of rise in the mould and a low teeming temperature, as used in these tests, the meniscus is bound to freeze near the interface, despite probable carbon pick-up from the dressing. With higher teeming rates and temperatures, the steel would probably 'roll' up the mould wall without any ripple formation caused by the bending of the meniscus in this manner.

Ripple formation is illustrated in Fig. 9, in which the steel surface is shown by:

a b c d	at time T
a g c′ d′	at time $T + \frac{1}{4}$ s
a g′ i c″ d″	at time $T + \frac{1}{2}$ s

The position of the boundary between solid and liquid steel can be calculated sufficiently accurately for this purpose from the equation $d = \sqrt{t}$, where d = solid layer thickness, in., and t = time, min. This boundary is shown as:

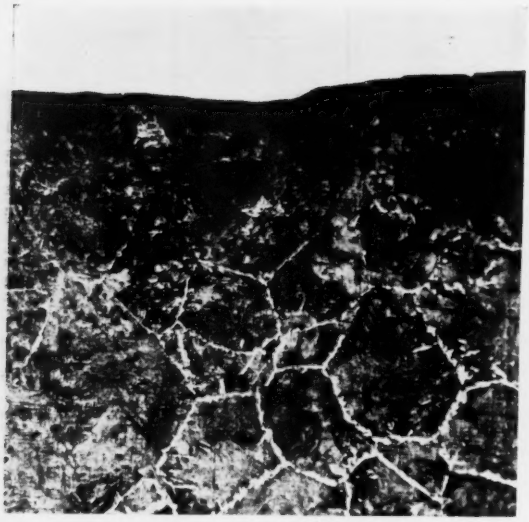


Fig. 13—Surface of ingot from mould coated with bituminous paint, showing carbon pick-up, series 2, cast 7 × 50

e f b	at time T
e′ f′ g	at time $T + \frac{1}{4}$ s
e″ f″ j k	at time $T + \frac{1}{2}$ s

The precise position of the boundary cannot be determined; the dotted lines f′hg and f″h′j show an estimate of its position, allowing for the reduced rate of chilling at the hollow of the ripple where there is a gap between steel and mould.

Cine film observations show that some flattening of the ripple occurs at about time $t + \frac{3}{4}$ s indicating that the solid layer of metal at g′ is very thin. Indeed, some of the ingots cast in moulds coated with thick layers of bituminous paint No. 1 showed that ' bleeding ' had taken place in the ripple hollows.

Laps—These, like ripples, appear to be caused by chilling of the steel meniscus. The effect is much more serious, however, and occurs when a crust of oxide, slag, or frozen steel forms over the meniscus, resulting in an over-lapping of the steel as illustrated in Figs. 11 and 12.

Volatile dressings considerably reduce the incidence of lap formation by keeping the meniscus ' open ' and free from oxide.

The factors favourable for the prevent of laps are:

(i) High teeming temperature
(ii) High teeming rate
(iii) Use of volatile dressings.

It is noteworthy that of the dressings used in these tests, alumina cement was the worst with regard to lap formation. The films showed that a slag-like product, formed by interaction between the molten steel and the alumina, produced a crust over the meniscus with consequent over-lapping of the steel.

Ingots cast in a nitrogen atmosphere gave noticeably less lapping with non-volatile dressings than those cast in air, which indicates that the oxidation of the meniscus is an important factor.

Cracks—Ingot surface cracks are generally caused by restriction in the mould, or by localized high surface stresses set up by differential cooling rates.

The prevention of surface cracks requires teeming conditions such that:

(i) There is no restriction of ingot shrinkage on solidification
(ii) The ingot skin builds up strength sufficiently quickly to withstand the internal ferro-static pressure
(iii) The inner surface of the mould is smooth and offers a uniform chilling effect.

High teeming speeds and temperatures tend to increase the incidence of cracking because the ferro-static pressure increases rapidly and the chilling effect of the mould wall is reduced, resulting in a slower build up of strength in the initial ingot skin. Hence, low teeming speeds and temperatures are necessary to reduce cracking, as indicated in Fig. 1. The reduction of cracking, therefore, calls for a volatile dressing in order that lapping will not occur with the low teeming speeds and temperatures used. This is particularly true with killed steels, in which the ingot skin is less able to withstand high stresses than the more oxidized steels, and lapping is also more prevalent.

Surface Pitting—' Ingot pitting ' is caused when gases are evolved between the ingot and the mould below the interface. This is likely to occur when high teeming speeds are used in conjunction with a thick layer of volatile dressing, in which case all the volatile matter is not driven off above the interface and the steel over-runs the dressing.

Carbon Pick-up at the Ingot Surface—Photomicrographs of sections etched with 2% nital (Figs. 13–17) show the effect of the dressings used on the sub-surface structure of the ingots.

In general, the volatile and charcoal based dressings produced definite carburization of the sub-surface, the non-volatile dressings and the non-dressed moulds gave decarburization. This strengthens the view that carburization occurs over the interface by

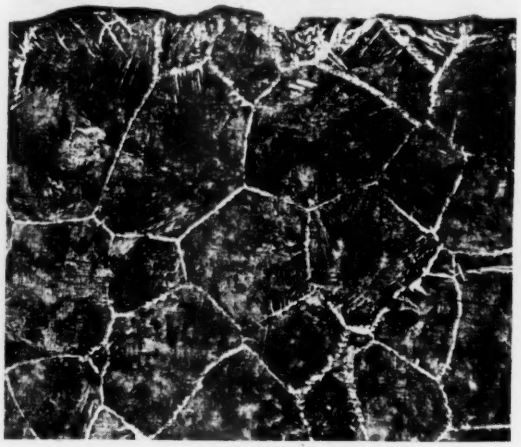


Fig. 14—Surface of ingot from tar reeked mould, showing decarburization, series 2, cast 7 × 50

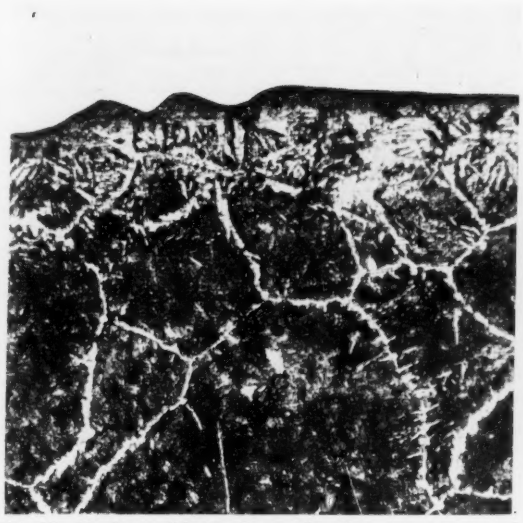


Fig. 15—Surface of ingot from alumina cement coated mould, showing decarburization, series 2, cast 6 × 50

gaseous reaction and, as shown later, this could result in the surface flow observed with the use of bituminous paint, and a reduction in the melting point of the steel surface.

In each case, the mould wall was stripped from one face of the ingot within a minute of finishing teeming. The other ingot face was allowed to cool in contact with the mould wall. Where decarburization occurred there was very little difference between the quickly and slowly cooled surfaces, and this indicates that any decarburization occurs mainly during or immediately following teeming.

It is possible that, as the dressing vaporizes just ahead of the interface, the steel meniscus absorbs carbon from the reducing gases, causing a localized

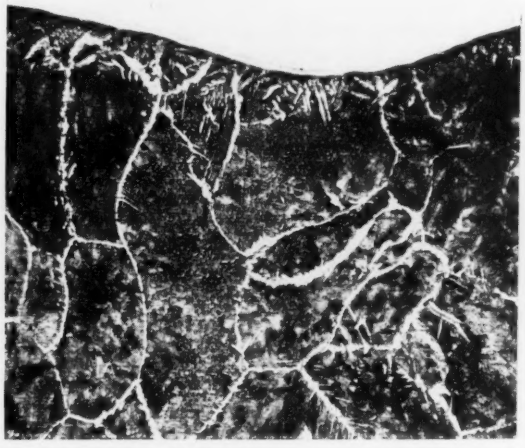


Fig. 16—Surface of ingot from non-coated mould, showing decarburization, series 2, cast 7 × 50

Fig. 17—Surface of ingot from mould coated with charcoal based dressing, showing cementite network due to carbon pick-up, series 2, cast 5 × 50

reduction in surface tension of the steel surface close to the interface. This would give rise to a surface flow away from the interface as noted in the films.

Figures showing the carbon pick-up at the surface of a small ingot obtained from a mould coated with a charcoal based dressing are given in Table XI. The other volatile dressings resulted in less carburization.

The carburization shown here is probably much more than would be found on a commercial ingot since the 2–3 mm thickness of dressing used may be excessive for the small test ingot. Moreover, commercial ingots remain hot for a sufficient time for further diffusion and for some surface decarburization to occur. It could be of importance in ingots that are machined before working, in that a harder surface layer would be obtained, but for ingots not so treated, a large part of the carburized layer would be scaled off in the soaking pits.

ADVANTAGES AND DISADVANTAGES OF CERTAIN DRESSINGS

Bituminous Paint No. 1 and Tar—Applied thinly, the bituminous paint did not appear to be very effective in keeping the surface oxide film away from the mould/metal surface. Slight carbon pick-up

Table XI

ANALYSIS OF SAMPLES PLANED FROM SURFACE OF INGOT CAST IN MOULD COATED WITH CHARCOAL BASED DRESSING

Depth of sample, in.	Carbon, %
0·0 – 0·05	1·06
0·05 – 0·10	0·89
0·10 – 0·15	0·72
0·15 – 0·20	0·67
0·20 – 0·25	0·62
0·25 – 0·30	0·65
0·30 – 0·35	0·62
0·35 – 0·40	0·61

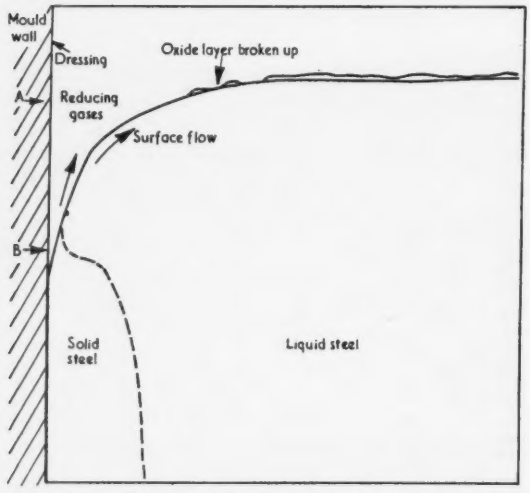


Fig. 18—Effect of bituminous paint No. 1 applied thinly (0·1–0·2 mm)

occurred, however, and the ripples on the ingot surface were typical of those usually obtained with a volatile dressing. The rise of the metal in the mould at teeming speeds from 12½ to 25 in./min was smooth and fairly even. No reaction could be seen at the interface.

Bituminous paint No. 1, applied in medium thickness of about 0·1–0·3 mm, gave conditions in which the steel rose smoothly and evenly up the mould wall over the range of teeming speeds obtained in the tests. No turbulence or reaction could be seen occurring at the interface except for a flow over the steel meniscus normal to the mould wall. This is illustrated in Fig. 18; the flow was effective in keeping the oxide film on the surface of the liquid steel away from the interface. There are two possibe explanations of this flow:

(i) Local reductions in surface tension of the steel near the interface
(ii) Propulsive effect of gas evolution.

The former seems the more reasonable explanation as no turbulence was noted. Fumes from the dressing could be seen above the steel, and in the film taken at 64 frames/s, the dressing could be seen melting and apparently flowing up the mould wall just ahead of the steel meniscus. This effect was probably an illusion caused by the vaporization of the dressing. Medium to thick coats (above 0·3 mm) of bituminous paint No. 1 resulted in a vigorous reaction below the steel surface. The ingots obtained showed a slightly greater degree of carbon pick-up but the surfaces were very similar to those obtained with thin coats of the dressing.

Applied more thickly, bituminous paint No. 1 gave rise to a violently propulsive reaction at the interface, and the occurrence of surface pitting was increased.

From a comparison of the physical and chemical characteristics of bituminous paints Nos. 1 and 2 with those of a coal tar based dressing, it is clear that in application to the moulds the bituminous paints offer the following advantages over the coal tar dressing:

(i) They contain no water so do not need dehydrating
(ii) They are more mobile and hence do not require heating, and give a thinner, more even coat
(iii) They have an appreciably shorter drying time
(iv) They evolve less pungent fumes in drying.

To some extent offsetting these advantages, the coal tar has a higher flash point, and may be less likely to cause an explosion if hot moulds are being dressed.

It is evident from the distillation curves that no volatile dressing should be applied to a mould at too high a temperature, otherwise excess volatile constituents are evolved, and the dressing becomes cokey in character.

During teeming, the volatilization of the bituminous paint occurs at a lower temperature than for the coal tar dressings. This is beneficial in creating a reducing atmosphere in the mould and renders the metal less likely to over-run the dressing, with consequent gas evolution below the interface, and surface pitting. This is only likely to occur, however, when the dressing is applied too thickly or too high teeming speeds are used.

In general, it is thought that bituminous paint No. 1 or tar applied about 0·1 mm thick gives the best results. With this thickness no serious defects occurred, and the induced meniscus flow ensured that no slag or oxide was entrapped in the ingot skin. It is probable that higher teeming speeds and temperatures than those used in these series of tests would eliminate ripples and give a smooth, defect-free ingot surface.

As an indication of the quantity of dressing required for a thickness of 0·1 mm to be applied to the mould wall, 1 gal of bituminous paint would be sufficient to coat about 10 four-ton moulds.

Alumina Cement—In spite of its heat insulating properties, alumina cement does not provide a smooth rise of steel in the mould. It gives no protection against oxide or slag entrapment in the ingot surface and the jerky rise of the steel, when low teeming temperature and speeds are used, gives rise to laps. Although ripple formation is inhibited, the surfaces obtained are rough and uneven.

When alumina cement was used in conjunction with an oxygen-free nitrogen atmosphere, a material of higher emissivity than liquid steel could be observed forming at the interface and spreading over the meniscus. This was almost certainly hercynite and was responsible for the high incidence of lapping with alumina cement dressing.

Soot (*Tar Reeking*)—It was thought that a layer of soot would provide a certain degree of heat insulation and also prevent surface decarburization. In fact, the results obtained showed very little difference between a tar reeked mould and a clean non-dressed mould.

Moulds coated with soot showed a greater tendency to give lapped ingots at low teeming speeds and temperatures than did moulds coated with volatile dressings. The meniscus was covered with oxide throughout the teeming, and the ingot surfaces were decarburized to about the same extent as those cast into non-dressed moulds, as shown in Figs. 14 and 16.

Table XII
DRESSINGS RECOMMENDED FOR PREVENTION OF DEFECTS

Defect	Cause	Conditions Necessary for Prevention	Dressings Recommended in Order of Preference
Cracking	Rupture of skin when stresses increase at a greater rate than the building up of the strength of the ingot skin just below the interface	Low teeming speed Low teeming temperature in conjunction with dressing	Volatile—This enables a lower teeming speed and temp. to be used without detrimental increase in lapping
Lapping	Presence of oxide or slag over meniscus preventing smooth rise of steel	High teeming speeds High teeming temperature in conjunction with non-volatile dressing, or medium teeming speeds and temperatures in conjunction with volatile dressing	Volatile Non-volatile
Splash and double skin	Freezing of steel splashes or surge near base of mould	Prevention of sticking of splashed steel	1 Charcoal based dressing 2 Volatile 3 Non-volatile
Bleeding	Steel breaking through ingot skin due to ferrostatic pressure building up too fast, or too slow chilling of ingot skin; mould too hot	Low teeming speed Low teeming temperature Low mould temperature	1 Non-volatile 2 Volatile (applied thinly)
Surface inclusions	Presence of slag or oxide over meniscus	Prevention of oxidation of steel surface. Keeping meniscus clear of slag particles	Volatile
Surface pitting	High teeming speed with excess dressing or volatile constituents on mould wall. Dressing not completely dried	Low teeming speed High teeming temperature	1 Non-volatile 2 Volatile (applied thinly)

When soot was used as a dressing in a mould through which oxygen-free nitrogen was passed throughout the teeming, the meniscus was almost clear of oxide, the metal rise appeared to be smoother and a slight surface flow over the meniscus away from the interface could be seen. The incidence of lapping was considerably reduced, indicating that the oxidation of the meniscus to some extent controls the tendency to lap formation.

Stearic Acid—This wax was included in the trials as a dressing which would volatilize without leaving any residue, in contrast to the bituminous paint. Only one cast was made using it. The film obtained showed that although some surface flow over the steel meniscus did occur, it was not fully effective in keeping the meniscus clear of oxide. The rise of the steel in the mould was fairly smooth and even, and the surfaces obtained were very similar to the ingots cast in moulds coated with bituminous paint No. 1.

Stearic acid, which has a melting point of 67° C, melted well above the steel so that the thickness on the mould wall could not be estimated with any degree of certainty.

Slight carburization of the ingot surfaces occurred.

Charcoal Based Dressing—This dressing gave rise to a violent turbulent reaction at the interface but the ingot surfaces obtained were quite smooth and free from defects except for slight pitting. Considerable carburization occurred (*see* Fig. 17) and in some cases this may be an undesirable feature.

CONCLUSIONS

The properties in a mould dressing found in this work to be important are:

(i) Volatility, preferably such that reducing gases are evolved just above the steel surface. It is important that evolution should not take place below the steel surface
(ii) The temperature of vaporizing of the dressing should be such that under normal works conditions only a proportion of the volatile constituents are evolved in application to the mould
(iii) A non-volatile base for the dressing to prevent initial contact between steel and mould wall
(iv) The dressing should be non-oxidizing and should not react with the steel to give any slag or oxide constituents
(v) The dressing should be in a form favourable for easy application to the mould wall
(vi) The drying time should be short
(vii) Excessive fumes should not be evolved
(viii) The flash point should be high enough to prevent danger of explosion in application.

The types of dressing recommended for the control of the principal ingot surface defects are shown in Table XII.

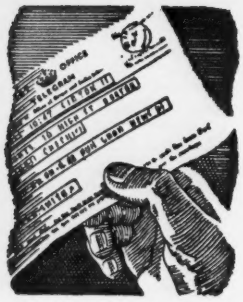
Of the main types of mould dressing generally available, the volatile materials offer the most advantages, and, taking into account ease of application and efficient functioning of the dressing, the bituminous paints appear to be the best. Coal tar and charcoal based dressings are just as effective in reducing ingot surface defects but are a little more troublesome in application.

The volatile dressings are most effective for the following reasons:

(i) They prevent oxidation of the steel meniscus
(ii) They keep surface films or slag away from the interface
(iii) They prevent excessive decarburization of the ingot surface
(iv) They prevent the sticking of splashed steel to the mould wall
(v) They promote a smooth metal rise in the mould.

Acknowledgments

The author is greatly indebted to Dr. J. Pearson, Mr. R. Mayorcas, Mr. W. H. Glaisher, and to members of the Ingot Moulds Sub-Committee for guidance; to Mr. J. Ravenscroft for advice and help with the instrumentation; to Mr. A. W. McCrum who was responsible for most of the mathematics; and to Mr. P. Hobson, Mr. H. Leaper, and other members of the staff of B.I.S.R.A. who assisted with the experimental work.



N E W S

Announcements and News of Science and Industry

NEWS OF MEMBERS

Dr. **Charles Sykes,** F.R.S., Member of Council, Bessemer Gold Medallist 1956, Managing Director of Thos. Firth and John Brown Limited, has been created a C.B.E.

Mr. **G. L. Bailey,** C.B.E., Director of the British Non-ferrous Metals Research Association, has been elected President of the Institution of Metallurgists.

Mr. **W. E. Ballard** has been elected a Vice-President of the Institution of Metallurgists.

Mr. **W. E. Bardgett** has been elected to the Council of the Institution of Metallurgists.

Colonel **J. M. Bevan,** D.L., M.C., J.P., has been appointed to the Board of Duport Ltd., consequent on its acquisition of the Briton Ferry Steel Company.

Mr. **E. J. Bradbury** has been elected to the Council of the Institution of Metallurgists.

Mr. **D. Bruce-Gardner,** Director and General Works Manager of John Lysaght's Scunthorpe Works Ltd., has been appointed a Joint General Manager.

Mr. **J. M. Couch** has taken up an appointment with Stewarts and Lloyds Ltd., Bilston.

Captain **H. Leighton Davies,** C.B.E., J.P., Past President of the Institute, has been appointed Sheriff for Carmarthenshire.

Dr. **R. A. Dodd** has resigned from the staff of the Department of Mines, Ottawa, and has been appointed Assistant Professor of Metallurgy in the University of Pennsylvania, Philadelphia, Pa.

Mr. **A. B. FitzJohn** has taken up an appointment as Assistant to the Chief Engineer with William Jessop and Co., Ltd.

Mr. **M. G. Gemmill** has left the Research and Development Department of the United Steel Companies Ltd., and has taken up an appointment as Chief Metallurgist to the Central Electricity Authority.

Mr. **J. G. Haenel** has been appointed a Director of the Companhia Siderurgica Paulista, São Paulo, Brazil.

Mr. **J. G. Hines** has been awarded the degree of Ph.D. in Cambridge University.

Dr. **N. P. Inglis** has been elected to the Council of the Institution of Metallurgists.

Mr. **W. L. James,** Member of Council of the Institute, Managing Director of John Lysaght's Scunthorpe Works Ltd. for the past eight years, is retiring from the iron and steel industry owing to ill health.

Mr. **K. R. Jason** has relinquished his post as Senior Metallurgist with Quasi-Arc Ltd. and has been appointed Chief Metallurgist with Joseph Sankey and Sons Ltd., Bilston.

Mr. **F. H. Keating** has been elected to the Council of the Institution of Metallurgists.

Mr. **J. N. Kilby** has been appointed to the Board of the Briton Ferry Steel Company.

Dr. **K. V. Kiukkola** has taken up an appointment with Outokumpu Oy at their copper smelter in Harjavalta, Finland.

Mr. **L. E. Leake** has taken up an appointment with the Chemistry Department of the British Iron and Steel Research Association.

Mr. **D. B. Manwaring** has left the Electrical Engineering section of the Plant Engineering Division of B.I.S.R.A. to take up an appointment with the Ministry of Supply, Armoured Fighting Vehicles Research and Development Establishment, Chertsey.

Mr. **James Mitchell,** C.B.E., Past President and Bessemer Medallist of the Institute, has been elected Senior Vice-President of the Institution of Metallurgists.

Professor **A. J. Murphy** has been elected a Vice-President of the Institution of Metallurgists.

Miss **Dorothy Pile** has taken up the appointment of Technical Information Officer to the British Jewellers' Association and is in charge of the recently formed Technical Department.

Mr. **T. Powell** has been appointed Managing Director of British Acheson Electrodes Ltd., Sheffield.

Mr. **J. Ravenscroft** has been awarded the James Prescott Joule Commemorative Medal for a paper on the efficient utilization of energy in the electric arc furnace. The silver medal is awarded annually by the Sir W. H. Bailey Trust for the best paper on energy transformation submitted from young science graduates in the Manchester area.

Mr. **A. L. Sutton** has relinquished his post at the R.T.S.C. Laboratories, Aylesbury, and has taken up an appointment in the Metallurgy Department of the Research Laboratories of the General Electric Co., Ltd., Wembley.

Dr. **P. A. Young** has left the Imperial Smelting Corporation to take up an appointment as Manager, Research and Development, to the Head, Wrightson Group.

Obituary

Mr. **James Spencer Hollings, C.B.E.** (*elected* 1903), Honorary Vice-President and Honorary Member, Bessemer Gold Medallist 1946, on 21st June, 1956.

Major **Gerald Rhodes Rosevere,** M.B.E. (*elected* 1896; *life member* 1948), on 4th February, 1956.

Mr. **Walter C. Smith** (*elected* 1939), Director of the Guest Keen Iron and Steel Co., Ltd., on 3rd March, 1956.

CONTRIBUTORS TO THE JOURNAL

Professor **Paul G. Bastien, D.Sc.**—Director of Research at the Laboratoire de Physique des Métaux, École Centrale, Paris.

Professor Bastien was born in 1907 and was educated at the École Centrale des Arts et Manufactures, being awarded the Doctorat ès sciences physiques and the Doctorat de Droit.

He has worked under Professor Albert Portevin as scientific adviser to the Air Ministry in the metallographic laboratories of the École Centrale. His initial researches

P. Bastien

were concerned with light alloys, the cooling of metals and alloys, and forgeability. He has pursued parallel careers in industry and education, specializing in metallurgy and metal physics.

In industry, Professor Bastien was Ingénieur-Conseil of the Établissements Schneider from 1936 to 1946, becoming Directeur Scientifique in 1946 and holding a similar position at the Société des Forges et Ateliers du Creusot from 1950. In education, he was appointed Professeur de Physique Générale et de Physique des Métaux at the École Centrale in 1942 and became Director of Research in 1953. He also teaches at the Centre d'Études Supérieures de la Sidérurgie.

The researches of Professor Bastien and his collaborators are well known and have formed the subject of numerous papers published in France and England. Their merit has been recognized by awards from the Académie des Sciences and the Société des Ingénieurs Civils de France; the Société d'Encouragement pour l'Industrie Nationale and the Association Technique de Fonderie have awarded him their Gold Medals.

Professor Bastien has been President of the Comité International des Méthodes d'Essai des Fontes (1951–55), of the Conférence des Ingénieurs de l'Europe, du Commonwealth, et de l'Union Panaméricaine (Brussels, 1954), of the Association Technique de Fonderie (1945–48), of the Société des Ingénieurs Soudeurs (1951), of the Société des Ingénieurs Civils de France (1945), and of the Union des Associations Scientifiques et Industrielles Françaises (1954).

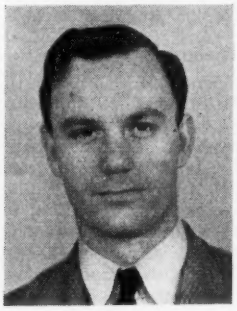
C. J. Thwaites, B.Sc., A.R.S.M., A.I.M.—Assistant Chief Metallurgist with the Tin Research Institute.

Mr. Thwaites was educated at Sutton County Grammar School and graduated from the Royal School of Mines with an honours degree in metallurgy in 1948. From 1948 to 1952 he was initially Bursar and subsequently investigator at the British Non-ferrous Metals Research Association, working on the creep and fatigue properties of lead alloys. Since 1952 Mr. Thwaites has been with the Tin Research Institute working on various aspects of tinplate, hot tinning, and soldering.

J. H. Welch, M.A.—Senior Scientific Officer at the Building Research Station of the Department of Scientific and Industrial Research.

Mr. Welch was born in Bradford in 1925 and was educated at Stowe and at King's College, Cambridge, where he obtained a state bursary to study Natural Sciences for two years.

He joined the Building Research Station in 1945, where he is now a Senior Scientific Officer and Head of the section studying the constitution of cement and slags and related problems. Mr. Welch was recently awarded a Bowen Prize by the Institute of Physics for a paper on high-temperature microscopy.



C. J. Thwaites

J. H. Welch

BRITISH IRON AND STEEL RESEARCH ASSOCIATION

Electronic Computor

It has been decided that B.I.S.R.A. should purchase a Ferranti "*Pegasus*" digital computor, to be installed during the first half of 1957.

Open Days at Sheffield Laboratories

The Association's laboratories at Hoyle Street, Sheffield, will be open to members on Thursday and Friday, 6th and 7th September, 1956. The work of the Steelmaking, Mechanical Working, and Metallurgy Divisions will be displayed, providing a review of progress made since the laboratories were opened in November, 1953. There will also be smaller displays of the work done in London, at Swansea, and at Normanby. In addition to the exhibits there will be demonstrations and short films.

Forty-sixth Steelmaking Conference

The 46th Steelmaking Conference was held at Ashorne Hill on the 2nd and 3rd May, 1956. Mr. R. W. Evans of the Steel Company of Wales Limited, Chairman of the Steelmaking Divisional Panel, was to have presided, but was unable to be present. His place was taken by the Vice-Chairman, Dr. J. H. Chesters of the United Steel Companies Limited, who is Chairman of the Steel Practice Committee.

The subject of the first session was the use of models for investigating the design and operation of O.H. furnaces. Mr. W. P. Cashmore spoke on the application of models at the Guest Keen Iron and Steel Co., Ltd., and Mr. A. E. Pearson described experiments with a model furnace at Dorman Long (Steel) Limited.

A joint paper on model work at Ijmuiden in Holland was presented by Mr. E. M. Hubbard, Dr. J. van Langen, and M. Rivière of the International Flame Research Foundation, who dealt with the relation between cold model, hot model, and production furnace results. A description of the hot model calorimeter O.H. furnace at the Sheffield Laboratories and an account of some B.I.S.R.A. research was given by Mr. R. Mayorcas of the Steelmaking Division. There was a very lively discussion on these papers and Professor Thring took a prominent part.

In the second session inactive mixer linings were considered, and Mr. H. Parnham of General Refractories Limited gave a paper on refractory problems of hot metal mixers. Inactive mixer lining construction at Ebbw Vale and at Corby was described by Mr. H. F. Padbury of Richard Thomas and Baldwins Limited and Mr. D. Bird of Stewarts and Lloyds Limited, respectively.

Cutlery Research Council

The Cutlery Research Council, in agreement with B.I.S.R.A., has appointed its Research Superintendent, Mr. C. N. Kington, to be Director of Research. Mr. Kington, who is group manager of the B.I.S.R.A. Sheffield laboratories, has hitherto been seconded to the Cutlery Research Council in a part-time capacity. He will continue in that capacity as Director of Research.

Demolition of Cementation Furnaces

Extension to the Sheffield laboratories will necessitate demolishing two of the cementation furnaces. The third furnace will, however, be retained as a historical monument to the process which provided most of Britain's steel from early in the 17th century until the 19th century.

AFFILIATED LOCAL SOCIETIES

West of Scotland Iron and Steel Institute

The West of Scotland Iron and Steel Institute is arranging a one-day conference on the application of low-alloy steels for welded pressure vessels, to be held at 39 Elmbank Crescent, Glasgow, on 19th October, 1956. The following papers will be presented:

"*Development of Mn–Cr–Mo–V High-tensile Steel for Pressure Vessels*," by W. Barr and I. M. Mackenzie.

"*Important Considerations for the Welding of Cr–Mo Steels*," by O. R. Carpenter.

"*Welding of Large Pressure Vessels in Low-alloy Steels*," by Dr. H. Harris.

"*A Low-alloy Quenched and Tempered Steel for Pressure Vessels*," by J. M. Hodge and L. C. Bibber.

"*Some Principles of Design for Pressure Vessels and Boilers made of Low-alloy Steel*," by P. W. Kerkhof.

"*Principles of Fortiweld Steel*," by Dr. L. Reeve.

A full report of the Conference and discussion will be published in the *Journal of the West of Scotland Iron and Steel Institute*, and will also be available as a reprint.

Liverpool Metallurgical Society

The following Officers and Committee have been elected for the session 1956–57:

Officers

President	W. ROSE
Senior Vice-President	E. DAVIS
Vice-President ..	J. S. W. GRAHAM
Honorary Secretary..	J. F. GIBLIN
Honorary Treasurer..	B. TODD

Committee

C. Bodsworth, G. T. Callis, R. Cobill, H. J. Miller, B. Ryan, and A. Ward, with the addition of A. E. Griffin (*Retiring President*), R. S. Brown, H. Edwards, B. L. Farthing, and S. J. Kenneth (*Honorary Vice-Presidents*).

EDUCATION

Expansion of Metallurgy at Oxford

At a meeting of Congregation on Tuesday, 12th June, a statute was promulgated which added the Science of Metals to the list of subjects that may be offered by candidates in the Honour School of Natural Science.

This subject covers much of the ground that is covered by those who offer chemistry in that Honour School. The amount of organic chemistry, however, has been reduced in order to provide room for the science of metals.

The new course is divided into Parts I and II on the same lines as the chemistry course, and in Part II a candidate will submit a thesis on a metallurgical subject after spending a year on research. Classified honours will be awarded on the combined results of both parts.

The new course marks a further stage in the development of metallurgy at Oxford which follows the establishment of the George Kelley Readership by the Pressed Steel Company and the appeal for funds for the subject which was strongly supported by the late chairman of the company, Major Albert Pam.

Post-graduate research work in metallurgy has been carried out at Oxford for more than 30 years under Dr. W. Hume-Rothery, O.B.E., F.R.S., in the Inorganic Chemistry Laboratory, where its development owes much to the interest of Professor Sir Cyril Hinshelwood, F.R.S.

An independent Department of Metallurgy under Dr. Hume-Rothery is now being set up in temporary accommodation, and it is hoped that a new laboratory will be built in the next few years.

The new development is particularly welcome in view of the close association of Oxford with metal-using industries.

NEWS OF SCIENCE AND INDUSTRY

Annual General Meeting of Institution of Metallurgists

The Annual Luncheon and Annual General Meeting of the Institution of Metallurgists took place on 15th May, 1956, at the Park Lane Hotel, London, W.1. The principal speaker, the Rt. Hon. Sir David Eccles, K.C.V.O., Minister of Education, referred to the part played by the Institution in furthering technological education and to the progress made by the Institution. The new President, Mr. G. L. Bailey, C.B.E., in his Presidential Address, discussed the important work of the Institution in promoting the interest of metallurgists and considered the future prospects for metallurgy, potential metallurgists, and for the Institution, of which the total membership is now 3331. He went on to consider the Institution's qualifications and the type of training which a metallurgist required, emphasizing particularly the need of broad training in the humanities and basic science, as a preliminary to specialist instruction in metallurgy.

Course on Air Pollution—Sheffield

The University of Sheffield Department of Extramural Studies in co-operation with the Yorkshire Council for Further Education is arranging a course on "*Air Pollution*," to be held at a University Hall of Residence from 24th to 27th September, 1956. The course is intended for people concerned with industrial plant, fuel, and domestic appliances and will deal with the sources, analysis, and elimination of air pollution. Full details are obtainable from the Director of Extramural Studies, The University, St. John's, Crookes Valley, Sheffield 10.

Recruitment and Training of Young People

The Committee set up by the National Joint Advisory Council, under the Chairmanship of Mr. Robert Carr, M.P., Parliamentary Secretary to the Ministry of Labour and National Service, wants evidence about the working of the apprenticeship system and other forms of training. A questionnaire is being sent to all employers' and workers' associations, but, in addition, the Committee invites any person or organization who wishes to do so to submit evidence.

Evidence should be sent in writing to the Secretary of the Committee, Mr. K. H. Clucas, Ministry of Labour and National Service, Ebury Bridge House, London, S.W.1, not later than 31st October, 1956. It would help if intention to submit evidence was notified not later than 31st July, 1956.

Bessemer Gallery at Science Museum

In co-operation with the Science Museum, South Kensington, the British Iron and Steel Federation is to create a new ferrous metallurgical display in the Museum to commemorate Sir Henry Bessemer. For this purpose the sum of £50,000 has been allocated. It is hoped that the gallery will be completed within three years.

The present space of the iron and steel section is 2800 ft^2 but even this is partly intermingled with the exhibits of the non-ferrous metallurgical industries. As part of their general scheme of reorganization, the Museum Authorities hope to make available 3500–4000 ft^2 in a self-contained and separate section.

Margam's Third Development Stage

The Steel Company of Wales has obtained approval from the Iron and Steel Holding Realization Agency to raise ingot output from 2·4 to 3 million tons per annum by 1960. The new scheme will increase the Company's sheet and tinplate capacity by 435,000 tons to about 2¼ million tons per annum, and forms an important part of the steel industry's plans to meet home demand and increase the volume of exports. The new steelmaking capacity will be oxygen-blown Bessemer.

Open Day at N.P.L.

An 'Open Day' was held at the National Physical Laboratory, Teddington, on Friday, 25th May, when the laboratories were open to inspection by scientists and technologists from all over the U.K. Among the special displays arranged by the Metallurgy Division was an exhibition of some of the results of the investigation into the properties of high-purity iron and iron alloys, which has been in progress for some years. Of particular interest were the recent developments in the study and preparation of single crystals of iron and the examination of the effect of texture in rolled bars. Also on display was an apparatus, still on a laboratory scale, for the elimination of sulphur in the preparation of high-purity iron, using ion-exchange membranes. Of more general interest were the demonstrations of electron microscope techniques and of the use of radioactive tracers in metallurgical investigations.

Increased U.K. Oil Consumption

In the booklet "*Statistics Relating to Consumption and Refining Production, 1954 and 1955*," published recently by the Petroleum Information Bureau on behalf of the U.K. Petroleum Industry Advisory Committee, it is shown that in 1955 oil consumption increased by more than 10% over the previous year. The total consumption of gas, diesel, and fuel oils by open-hearth steelmakers rose from 861,780 tons to 1,018,765 tons, of which 1,017,584 tons were of fuel oil, and total consumption of these fuels in other steelmaking processes rose from 482,866 tons to 549,934 tons.

Royal Belgian Society of Engineers

At the General Assembly of the Société Royale Belge des Ingénieurs et des Industriels, held on 22nd April, 1956, the retiring President, Monsieur Maurice Berger-Hainaut, was succeeded by Monsieur Robert van Cauwenberghe.

Conference on Human Problems in Industry—Oxford

The Duke of Edinburgh's Study Conference on the Human Problems of Industrial Communities within the Commonwealth and Empire, which takes place in Oxford from 9th to 27th July, will include group tours of industrial centres. Delegates visiting Sheffield will see the works of the English Steel Corporation Ltd. and Steel, Peech and Tozer.

Record Outputs from Appleby-Frodingham

Production of steel plates from the Appleby-Frodingham Steel Company's Appleby plate mills during the four weeks ending 14th April, 1956, was 36,022 tons; the previous record was 34,993 tons set up during the four weeks ending 26th December, 1953. During the week ending 14th April, 1956, also, the Seraphim sinter plant produced 43,270 tons, thus exceeding the previous best of 43,070 tons produced in the week ending 25th February, 1956. Output from this sinter plant over the four weeks ending 14th April was also a record at 162,410 tons.

Blast-furnaces for India

Head, Wrightson and Co., Ltd., are to design and manufacture three blast-furnaces, together with gas-cleaning plant and complete ore-preparation plant, for the Indian Government. The furnaces will have a hearth diameter of 27 ft, each capable of producing about 1000 tons of iron per day.

New Acheson Plant in the Netherlands

Acheson Colloids Ltd., of London, are to open a factory at Scheemda in the province of Groningen, Netherlands, which will manufacture fine dispersions.

Corrigendum

In the paper on "Corrosion Resistance of Some Austenitic Cr–Ni Steels of 18/8/Ti Composition" (March, 1956, issue of the *Journal*, p. 248), the caption to Fig. 5 should read "... steels *A* and *B* ..." and the sub-caption below the illustration should read "steel *B* after 7000h exposure."

Changes of Name and Address

The Anglo Metal Company Ltd. has removed to Garrard House, 31–45 Gresham Street, London, E.C.2 (MONarch 8800).

Firth Brown Tools Ltd. have changed the address of their Manchester Office to Speedicut House, Royle Street, Fallowfield, Manchester 14.

The Market Development Department of the British Iron and Steel Federation is now located at Steel House, Tothill Street, London, S.W.1 (telephone WHItehall 1030; telegrams Moresteel, Sowest, London).

John Cockerill S.A. and d'Ougrée-Marihaye S.A. have combined to form Cockerill-Ougrée S.A.

The Applied High-frequency Induction Heating Division of Wild-Barfield Electric Furnaces Ltd. has removed to Elecfurn Works, Watford By-pass, Watford, Herts.

The Minerva Detector Co., Ltd. has removed to Lower Mortlake Road, Richmond, Surrey.

DIARY

3rd–4th July—British Iron and Steel Research Association (Plant Engineering Division)—13th Conference—Theme: "*Works Transport*"—Sheffield.

9th–27th July—Duke of Edinburgh's Study Conference—"*Human Problems of Industrial Communities in the Commonwealth and Empire*"—Oxford.

11th–14th July—Institute of Physics (Non-destructive Testing Group)—Summer Meeting on "*Physics of some New Aspects of Destructive Testing*"—H. W. Wills Physical Laboratory, Bristol.

TRANSLATION SERVICE

(The previous announcement was made in the June, 1956, issue of the *Journal*, p. 230.)

Translations Available

No. 510 (German). H. A. Vogels: "Influence of Various Alloying Elements on the Properties of Heat-resisting Chromium–Nickel Steels in the Temperature Range 600–700° C." (*Stahl u. Eisen*, 1955, vol. 75, 5th May, pp. 559–570).

No. 511 (German). B. von Sothen: "Effect of Increased Demands on Output and Quality on the Design, Operation, and Control of Rolling-mill and Forge Furnaces." (*Stahl u. Eisen*, 1955, vol. 75, 2nd June, pp. 709–718).

ABSTRACTS OF CURRENT LITERATURE AND BOOK NOTICES

Iron and Steel Manufacture and Related Subjects

CONTENTS

	PAGE
Ores—Mining and Treatment	320
Fuel—Preparation, Properties, and Uses	320
Temperature Measurement and Control	323
Refractory Materials	323
Blast-Furnace Practice and Production of Pig Iron	324
Treatment and Use of Slags	325
Production of Steel	325
Production of Ferro-Alloys	326
Foundry Practice	326
Reheating Furnaces and Soaking Pits	331
Heat-Treatment and Heat-Treatment Furnaces	331
Forging, Stamping, Drawing, and Pressing	332
Rolling-Mill Practice	332
Machinery for Iron and Steel Plant	333
Lubrication	333
Welding and Flame-Cutting	334
Machining and Machinability	336
Protective Coatings	337
Properties and Tests	337
Metallography	338
Corrosion	338
Analysis	340
Industrial Uses and Applications	340
Historical	341
Miscellaneous	341
Book Notices	341
New Publications	344

ORES—MINING AND TREATMENT

Geochemistry and Radioactivity of Some Mn and Fe Bog Ores. P. Ljunggren. (*Geol. Föreningens Förh.*, 1955, **77**, Jan.-Feb., 33–44). [In English]. The trace element content of different bog ores has been measured and it appears that most of these elements are present in higher concentrations in the manganiferous rather than the ferriferous bog ores. Charts show that the radioactivity of the manganiferous ores is also higher.—G. G. K.

Sampling in Sedimentary Iron-Ore Deposits. H. Kolbe. (*Arch. Eisenhüttenwesen*, 1955, **26**, Apr., 183–197). The results of sample-taking in an iron-ore quarry and seven mines during the last 16 years are reported. The methods of sample-taking under the various conditions and at various stages, structural examination of the ore deposit, chemical analysis, and evaluation and recording of the results obtained are described in detail.

Post-War Development of the Iron Ore Mine Wohlverwahrt–Nammen. G. Glatzel and O. Hahn. (*Z. Erz. u. Met.*, 1955, **8**, Feb., 59–72). The mine works a very low-grade lime-rich sedimentary iron-ore. Full details are given of development, methods of working, haulage, and mine management. Operating cost statistics are quoted and costs of various methods of ore extraction are compared.—E. C.

Ore Blending at Shipping Dock Insures Uniform Blast Furnace Burden. M. W. Griswold. (*J. Met.*, 1955, **7**, Sept., Section 1, 956–959). The lack of uniformity of Minnesota ores necessitates the use of various methods at the Great Northern Ore Docks to produce a uniform shipping grade for blast-furnace use. A description is given of these methods and typical analyses are quoted.—G. F.

Improving the Planning of Sinter Plants. Ya. F. Chel'tsov. (*Stal'*, 1955, (3), 215–217). [In Russian]. The waste of effort involved in correcting badly planned sinter plants is indicated and suggestions are made for improving plant design.—S. K.

The Enrichment of Iron Ores. L. Coche. (*IRSID, Centre d'Etudes Supérieures de la Sidérurgie*, Metz, 1954, *Les Cahiers du Cessid*, No. 24, p. 157). Physical methods of upgrading ores are discussed in detail. Preparation by crushing and grinding, and classifier, jig, and table operations are outlined. Heavy-media separation, Humphrey spirals, and cyclones are also described. Magnetic separation, flotation, and electrostatic methods are also included. Chemical and metallurgical treatments include roasting of carbonate ores and magnetic roasting. Smelting methods include the low-shaft furnace, the rotary kiln, the Renn sponge-iron process, and the Wiberg process. Specific practice in Sweden, Norway, various parts of the U.S.A. and Canada, Germany, and other countries is surveyed, and methods of treating particular French ores are described. Numerous flow-sheets and operating data are given.—T. E. D.

The Sizing of Lump Ores. H. Pohl. (*Stahl u. Eisen*, 1955, **75**, Oct. 6, 1295–1300). The mechanical condition of lump ores can be divided into the following groups, according to size distribution. (1) Distribution into three grades: 0–5 mm = fine, 5–50 mm = pebble, 50–250 mm = lump. This conventional distribution should still be used when ore is hand-sampled and assessed visually. (2) Distribution into five grades: 0–1 mm = dust, 1–5 mm = fine, 5–50 mm = pebble, 50–120 mm = lump, 120–250 mm = coarse lump. This distribution is more exact and therefore desirable. It demands, however, a large sample, taken mechanically or automatically, and must be sized in a bank of sieves. It is recommended that these two distributions only should be used in technical and commercial considerations.—J. P.

Marmora Mine and Plant Ships Pellets to Feed Bethlehem's Blast Furnaces. (*Eng. Min. J.*, 1955, **156**, July, 75–79). Details are given of the open-cast iron-ore mine and pelletizing operations of Marmoraton Mining Co., Ltd., Ontario, a subsidiary of Bethlehem Steel Corp. Magnetite ore, averaging 37% Fe, is beneficiated to 65% Fe. Shipments have begun, and an output of 500,000 tons of pellets annually is planned.

A New Method for Characterising Separation Results in Beneficiation Processes. O. Sommer. (*Glückauf*, 1955, **91**, July 30, 878–889).

Agglomerating Iron Ore Concentrates. S. R. B. Cooke and T. E. Ban. (*Chem. Eng. Prog.*, 1955, **51**, Aug., 364–368). Pelletizing of magnetic taconite from the Mesabi is described, with information and discussion on the factors influencing balling, namely specific surface, nature of the additives, and mineralogical composition of the ore. Production of pellets by firing green balls is also examined, and the effects of firing temperature and atmosphere, and of additives on pellet strength, are noted.—T. E. D.

FUEL—PREPARATION, PROPERTIES, AND USES

Transient Heat Conduction in Multiphase Media. J. H. Weiner. (*Brit. J. Appl. Phys.*, 1955, **6**, Oct., 361–363). An

analytical semi-inverse procedure has been evolved for determining the problem of a semi-infinite mass of material initially at a uniform temperature, the surface of which is maintained at a different constant temperature, where the material may change phase an arbitrary number of times in passing from its initial to its final temperature.—E. E. W.

A New High Precision [Aneroid Bomb] Calorimeter for the Measurement of Heats of Combustion: and the Heat of Combustion of Succinic Acid. G. Pilcher and L. E. Sutton. (*Phil. Trans. Roy. Soc.*, Series A, 1955, **248**, May 12, 23–44).

The Combustion and Radiation Characteristics of Oil and Coke-Oven Gas Jet Flames. H. W. Thring. (*J. West Scotland Iron Steel Inst.*, 1953–54, **61**, 165–182). The author discusses the desired characteristics of an O.H. furnace flame and concludes from a study of a working model that (i) fuel consumption in melting can be reduced by increasing the bath coverage by the flame or the flame emissivity; (ii) the melting time will be reduced by the same means if the roof temperature is not already at the maximum safe value; and (iii) there is an optimum flame length for each stage of melting. The author then describes experiments at Ymuiden which have shown how the flame length for a given fuel input can be controlled by varying the jet momentum or using turn burners, and how the flame emissivity depends on the type of fuel, particularly the C/H ratio. The possibility of making coke-oven gas give a luminous flame by preheating the fuel is also discussed.—L. E. W.

Combustion of Solid Fuels: Flame Research for Industrial Applications. D. T. A. Townend. (*Iron Coal Trades Rev.*, 1955, **171**, Nov. 25, 1285–1292). The author describes the various forms of fuel–air systems, and discusses the chemical reactions and rate-determining processes of solid fuel combustion. Research work specific to the combustion of pulverized fuel is also considered. Finally, the experimental work at Ymuiden with oil, gas, and pulverized fuel firing, is outlined.—G. F.

The Study of Flame Radiation. M. W. Thring. (*Proc. Inst. Vitreous Enamellers*, 1952–53, 1953–54, **11**, 67–74). The work of the International Flame Radiation Committee at Ymuiden is described.—A. D. H.

Luminous Flame Radiation in Furnace Heat Transfer. M. W. Thring and C. Hulse. (*Iron Coal Trades Rev.*, 1955, **171**, Sept. 16, 679–682). The authors discuss current research work on the role of luminous flame radiation in furnace heat-transfer, directed by the International Flame Radiation Committee at Ymuiden. Performance and combustion mechanism trials are described, and the present state of knowledge is summarized.—G. F.

Instrumentation as an Aid to Fuel Efficiency. R. Clare. (*Instrument Practice*, 1955, **9**, Aug., 752–755).

The Thermal Conductivity of Fluids. A. F. Schmidt and B. H. Spurlock, jun. (*Trans. Amer. Soc. Mech. Eng.*, 1954, **76**, 823–830). A compensating-type hot-wire apparatus is described which is capable of measuring the thermal conductivity of gases, vapours, and liquids over a wide range of temperatures. Results on the thermal conductivity of air, liquid, and vapour are given.—D. H.

A Quick Simple Method for the Simultaneous Determination of the Thermal Conductivity, Thermal Capacity and Thermal Diffusivity of Solids. O. Krischer and H. Esdorn. (*VDI–Forschungsheft*, (450), 1955, B, 28–39). A disc method for the determination of thermal properties is described in which four series-connected electrical heaters are used. The advantage of the method is that the measurements are made before steady conditions are established, and thus the method is quick. This enables the investigator to test solids containing moisture without serious error due to drying.—J. R. P.

The Solution of Unsteady Heat Conduction Problems using the Laplace Transformation. H. D. Baehr. (*Forsch. Ingenieurwesens*, 1955, **21**, (2), 33–40). The Laplace transformation is defined and the technique of employing it is described. The application of the transformation technique to the solution of problems of unsteady heat conduction is then described. To illustrate the method, the temperature–time relationship with depth into a cold wall, with one face instantaneously raised to a given temperature, is established.—J. R. P.

Recent Infra-Red and X-ray Studies of Coal. J. K. Brown and P. B. Hirsch. (*Nature*, 1955, **175**, Feb. 5, 229–233). Approximately 75% of the carbon in coals is shown to be in the form of condensed aromatic units, and for a coal of 85% carbon these contain about four or five rings.—B. G. B.

On the Value of Raw Coal. A. Terra. (*Rev. Ind. Min.*, 1955, **36**, Feb. 1, 215–225). Owing to the increase of low-grade coals, methods for making the best use of them have been studied, including sorting, washing various-size fractions and slimes treatment. The use, both domestic and industrial, to which such prepared coals may be put is also considered.

Coke Ovens as Gas Manufacturing Plant. G. Dougill and A. C. Nelson. (*Inst. Gas Eng., Publ.* 471, 1955). The early investigations carried out before deciding to install two batteries of coke ovens at Beckton Works, London, in 1930 are described.—B. G. B.

Automatic Regulation of Heating on Two Babcock and Wilcox Boilers. Hemery. (*Centre Doc. Sidér., Circ. Inform. Tech.*, 1955, **12**, (10), 1989–1999). Details are given of a boiler plant supplying a 27,000-kW generator unit. Two boilers, each having a maximum capacity of 80 tons of steam per hour at 72 kg/cm² pressure, are fired separately or simultaneously by blast-furnace gas, coal on a moving grate, and coke-oven gas. The methods of burner and grate control are outlined and instrumentation is described in detail.—T. E. D.

New Power, Steam and Blowing Installations at the Ohio Steel Works. J. P. Katzenmayer. (*Iron Steel Eng.*, 1955, **32**, Aug., 109–117). This article describes new power equipment installed at Ohio Steel Works to replace old plant. Power, steam, and blast-furnace gas balances are given for conditions prior and subsequent to the installation of the new equipment. Details of the reasons which led management to adopt particular solutions are given.—M. D. J. B.

New Steam and Power Facilities—Ohio Steel Works, U.S. Steel. R. W. Worley. (*Iron Steel Eng.*, 1955, **32**, July, 99–101). Two new steam-generating units are described. These have each a guaranteed capacity of 250,000 lb/h of steam at 950 lb/in² at 900° F, and are arranged for burning blast-furnace gas.—M. D. J. B.

A Contribution to the Theory of Heat Transfer in Laminar Flow. M. V. Bodnarescu. (*VDI-Forschungsheft*, (450), 1955, B, 19–27). The heat transfer from a wall with a temperature discontinuity to a liquid in laminar flow is studied. The exact solutions lead to Whittaker functions for the two cases of an infinite flat wall and a circular cross-section tube. Allowance may be made in the solutions for a variation of wall temperature and the equations are developed for the case of a linear variation.—J. R. P.

A General Evaluation of Shell-and-Tube Heat Exchangers with Parallel and Cross Flow. A. F. Fritzsche. (*VDI-Forschungsheft*, (450), 1955, B, 5–18). The author uses a new method for establishing design criteria for shell-and-tube heat exchangers. The optimum performance with a given liquid may be obtained and the surface involved in the heat exchange made as small as possible.—J. R. P.

Evening Out the Properties of Coals in the Stockyard of a By-Product Coking Plant. M. G. Fel'dbrin, G. M. Dorogobid, V. L. Krol, and N. E. Baev. (*Stal*, 1955, (2), 103–106). [In Russian]. This is an account of a full-scale study of the effects of bedding and mixing on the constancy of properties, mainly ash content, of coal charges proceeding to coke ovens making blast-furnace coke. Stockyard bedding was found to be most effective and the provision of adequate space in all new plants is urged. Where part of the coal does not pass through the bedding system it is suggested that this should be added in constant proportion from separate bunkers.—S. K.

Researches on the Thermal Decomposition of Coal. M. F. Kessler. (*Paliva*, 1955, **35**, (10), 302–308). [In Czech]. The decomposition of coal as a function of temperature is studied in nitrogen. The thermogravimetric curves obtained are evaluated by a new method which facilitates differentiation between coals on the basis of the number of CH bonds which are lost above 600° C. The amount of volatile materials given up in the range 600–900° C, expressed as a fraction of the total weight of volatile fuel, is suggested as a new rational criterion for classifying coals. The gravimetric method used and the recording apparatus are described.—P. F.

The "Blofif" Heavy Liquid Process for Washing Fines. M. R. Blondelle. (*2nd International Conference on Coal Preparation: Rev. Ind. Min.*, 1955, **36**, Jan. 15, 192–205). In this process, a suspension of water and finely ground magnetite is used in a patented separator. It is generally used to treat coal less than 10 mm in size. The process is described in detail with descriptions of plants at present in operation. Magnetite consumption is of the order of 600–800 g/ton of coal treated.—G. E. D.

Removal of the 0·3–2 mm Fraction from the Freyming Washer by Cyclone Treatment in Clear Water. B. Bonnard. (*2nd International Conference on Coal Preparation: Rev. Ind. Min.*, 1955, **36**, Jan. 15, 206–214). A description of a cyclone plant at the Emibe Huchet power station producing 140 tons/day of washed coal fines with less than 5% ash. A flow sheet is given and each stage is fully discussed.—G. E. D.

Modern Coke Plant Now in Operation in Mexico. G. Bennett. (*Blast Furn. Steel Plant*, 1955, **43**, Sept., 1003–1005). A new coke-oven plant at Monclova in Mexico designed to manufacture 1694 tons of dry coke per day and consisting of two batteries of 57 ovens is described.—B. G. B.

Unusual Teething Troubles. T. W. Mills. (*Gas World*, 1955, **142**, Aug. 27, 478–481). Servicing of an 18 silica-oven coke battery during wartime conditions is described.—T. E. D.

New Coke-Oven Plant at Stanton: More Gas for the Grid. (*Iron Coal Trades Rev.*, 1955, **171**, Sept. 30, 795–796). The extension to the Hallam Fields coke-oven plant of Stanton Ironworks Co., Ltd., has recently been opened. Details are given of the coal-handling, blending, and crushing plant, the coke ovens, and the by-product units.—G. F.

Some Developments in Coal Carbonization. (*Gas Times*, 1955, **85**, Oct. 14, 21–30). Simon-Carves developments in coal treatment and carbonization plant are surveyed. Outlines of new coal-washing plant, coke-oven equipment, test coke-ovens, by-product plant, and flue-gas washing are given.

Trials with Six Cokes in the Blast Furnace. Ch. G. Thibaut, D. Sanna, B. Trentini, F. Douez, M. Pasquet, B. Menuet-Guilbaud, P. Vigneron, and R. Labourdette. (*Centre Doc. Sidér., Circ. Inform. Tech.*, 1955, **12**, (11), 2109–2143). The raw materials, method of carbonization, and physical properties of the cokes are given in detail. Comprehensive blast-furnace performance data for operation with each type are tabulated and compared. Relative coke rates are related to a coke property index derived from MICUM test results.

Contribution to the Knowledge of the Water Content of Metallurgical Coke. W. Tietze. (*Glückauf*, 1955, **91**, Sept. 24, 1094–1099). A statistical evaluation was made of the results of determining the water content of coke samples of various sizes, and varying in water content from 1·5% to 13·5%.

Study of the Precision of MICUM and IRSID Characteristics of a Batch of Coke. J. Ulmo and M. B. Menuet-Guilbaud. (*Institut de Statistique de l'Université de Paris: Journée de la Sidérurgie*, 1954, June 22, 3–11). Sampling methods of MICUM and IRSID are examined and compared statistically; 24-h production of a coke battery was used for the tests.

The Gasification of Carbon. J. D. Blackwood. (*Rev. Pure Appl. Chem.*, 1954, **4**, Dec., 251–274).

Gasification of Solid Fuels in a Fluidized Bed. P. S. Murthy and A. K. Das Gupta. (*J. Sci. Indust. Res.*, 1955, **14C**, Apr., 174–185). The authors describe tests to determine the effects of changes in the weight of fuel, and in the velocity, total rate of flow, and temperature. The differential rate of the $C–CO_2$ reaction with fuels at zero bed weight and the values of energy of activation for the reaction have been measured.

Producer Gas Evaluator. (*B.C.U.R.A. Quarterly Gazette*, 1955, (26), 11–13). A 'flame evaluator,' in which heat release from a flame can be determined, is very briefly described. Producer gas is supplied by a small producer, and a recuperator enables the effect of preheating the gas (up to 1000° C) to be studied. Some of the tar present in the gas cracks on preheating.—T. E. D.

The "Aérojet-Venturi R.B." Process for Gas Cleaning. R. M. G. Boucher and G. Vert. (*Génie Chem.*, 1955, **74**, July, 1–18; Aug., 38–50). A detailed account of the principles and operation of a Venturi gas washer is presented. Diagrams of typical arrangements and tables of factors used in calculation of the design are given. A number of installations of this type of gas cleaner are described with illustrations.—B. G. B.

Cleaning High-Pressure Blast-furnace Gas. E. N. Teverovskiĭ, M. M. Zaĭtsev, Yu. P. Mineev, Yu. A. Skoretskiĭ, G. I. Slastenkov, and P. S. Khomutinnikov. (*Stal*, 1955, (2), 172–179). [In Russian]. An experimental installation for cleaning gas from blast-furnaces on high top-pressure operation is described and test results are reported. The installation consisted of a cord-filled scrubber, an electrostatic precipitator, a turbulent gas washer (a pipe with a Venturi constrictor into which water is injected), and a cyclone for removing droplets. It reduced the dust-content of the gas to 10 mg/m³ with a water consumption of 1 l./m³ (both N.T.P.).

The Measurement of Fluid Flow by Means of a Micro-Venturi. V. Litchinko. (*Centre Doc. Sidér., Circ. Inform. Tech.*, 1955, **12**, (11), 2189–2204). The profile of a small Venturi, 90 mm dia. × 220 mm long, is defined. The behaviour of the micro-Venturi is considered theoretically in detail. Its calibration characteristics are given from experimental results. Its method of use in pipe-lines for dirty and wet gas is also mentioned.—T. E. D.

Considerations on the Economic Diameter of Supply Pipes for Blast Furnace Gas. J. Szczeniowski. (*Centre Doc. Sidér., Circ. Inform. Tech.*, 1955, **12**, (10), 2007–2011). The factors necessary for the calculation are listed, and the cost of transport per ton and per kilometre is estimated in general terms. Some results are shown graphically.—T. E. D.

Fuel Oil for Process Gas. (*Brit. Steelmaker*, 1955, **21**, Aug., 266–268). A description is given of a new process for converting heavy fuel into furnace gas by means of a non-atomizing oil cracker-gasifier. The process has been developed in France by the Office Central de Chauffe Rationelle and it is claimed that, in combustion, sulphur attack is less than that from an orthodox burner.—G. F.

Coal Preparation Plant at Nantgarw. (*Iron Coal Trades Rev.*, 1955, **171**, July 1, 38–42). The new coal preparation plant at Nantgarw Colliery treats the run-of-mine output in two processes, the heavy-medium Chance process and froth flotation.—G. F.

More Coke for Pig Iron. Extensions to the Stanton Ironworks Plant. (*Engineering*, 1955, **180**, Oct. 7, 512–513).

Post War Developments in Coke Ovens. (*Gas Times*, 1955, **84**, Sept. 2, 253–256).

Metallurgical Coke. F. Pintado Fe and J. R. Garcia-Conde. (*Inst. Nac. Carbon Bol. Inf.*, 1955, **4**, July, 169–181). [In Spanish]. The coking properties of 33 blends of three coals, a coking coal, a high volatile-matter caking coal, and a low volatile-matter non-caking coal were determined by the box-test method and plotted in ternary diagrams in order to evaluate the most beneficial blends. The coke with the best mechanical properties (utilization index 1·027) contained 63% of the coking coal and 10% of the low volatile-matter coal.—P. S.

Rheological Properties of Coke during Carbonization. D. Fitzgerald. (*Nature*, 1955, **175**, Mar. 19, 515–516). The strain retardation and stress relaxation phenomena of coal subjected to alternate application and removal of stress during constant temperature carbonization has been studied.—B. G. B.

Metallurgical Coke 1939–1955. J. Taylor. (*Iron Steel*, 1955, **28**, Sept., 431–435; Oct., 469–474, 479). The author first reviews the requirements of metallurgical coke used in the blast furnace, considering its chemical and physical properties, and then deals with the requirements of foundry coke for cupola use. Coke production is next discussed, and attention is given to the required properties of the coal, particularly in the plastic range. Finally, carbonizing conditions are described. The two parts contain respectively 65 and 68 references.—G. F.

Quality Control and Quality Specifications for Coke and Coke Breeze in Norwegian Iron Foundries. (*Tidsskr. Kjemi, Bergvesen Met.*, 1955, **15**, (1), 13–17). [In Norwegian]. Extracts are given from answers to a questionnaire sent to 15 Norwegian foundries concerning quality control methods in use. Sections deal with sampling, chemical and physical analyses, average analysis values obtained, characteristics desirable in an ideal coke, importance attached by the various foundries to coke quality and its influence on production costs, and the likelihood of increased use of Svalbard coal.

Studies of Size-Distribution Patterns and Breakage Processes for Metallurgical Cokes. J. B. Gayle and H. S. Auvil. (*U.S. Bur. Mines Rep. Invest.*, 5096, 1954, Dec.). A detailed study of the breakdown of metallurgical coke during shatter tests and tumbler tests has been carried out.—B. G. B.

Gas Producers: Design and Performance of New Two-stage Machine. (*Iron Steel*, 1955, **28**, July, 359–361). Details are given of the design and operation of the G.I. two-stage predistillation gas producer, installed in this country by International Furnace Equipment Co., Ltd. The production of cold clean gas, hot raw gas, and hot detarred gas is discussed, and the economics are considered.—G. F.

Gas and Destructive Distillation. C. H. Lewis. (*Soc. Chem. Ind., Reports on Progress of Appl. Chem.*, 1954, **39**, 181–189). The development of methods for the complete gasification of mined coal is reviewed. Mention is also made of the 'Carrier Gas' process and other methods of carbonization, the preparation of carburetted water gas and oil gasification, coal blending, and the use of natural gas. (34 references).—E. E. W.

Electric Furnace Ferrosilicon Fume Collection. L. Silverman and R. A. Davidson. (*Trans. Amer. Inst. Min. Met. Eng.*, 1955, **203**; *J. Met.*, 1955, **7**, Dec., 1327–1335). The authors summarize the results of a two-year study of methods of collecting ferro-silicon furnace fume. Studies were made with a wet unit using pneumatic and high-pressure fog nozzles in a cyclonic scrubber, and with dry units using wool-felt and fibre-glass with dilution air cooling, water-spray cooling, and direct filtration with high-temperature fumes. The performances and operating requirements of the two types are compared in detail, and the tests show that the dry unit shows most promise for direct application to this type of furnace.—G. F.

The Efficiency of Cyclone Dust Catchers. J. Dauphin. (*Génie Chim.*, 1955, **73**, May, 121–129). A method of deriving theoretically the efficiency of this type of dust catcher is described.—B. G. B.

Mechanical Dust Extraction Methods. G. Esche. (*Technik*, 1955, **10**, Nov., 669–673). Various types of cyclone dust extractors are described, with particulars of their performance. Ultrasonic and Cariolis extractors are also briefly referred to.

A Note on Multi-Stage Gas Washing. R. Long. (*Chem. Process Eng.*, 1955, **36**, Sept., 324–326). The author discusses the theoretical principles involved in the functioning of an absorption tower and of a multi-stage gas washer and thus shows that both are essentially the same concept. (8 references).—L. E. W.

Determination of the Coefficient of Pressure Loss in Pure Gas Pipelines. J. Szczeniowski. (*Centre Doc. Sidér., Circ. Inform. Tech.*, 1955, **12**, (10), 2005–2006). Factors affecting pressure losses in pipelines are discussed and a formula relating $\Delta\rho$ to pipe diameter, density of gas, speed and coefficient of friction is given. The effect of variations in the equivalent roughness 'κ' on the coefficient of friction 'λ' is tabulated.

The Spherical Gas Holder for the Communal Gas Works at Heerlen, (Holland). H. G. Noordman and G. van Schaick Zillesen. (*Lastechn.*, 1955, **21**, Aug., 127–130). [In Dutch]. This article is a brief description with several illustrations of the first welded spherical steel gas holder to be constructed in Holland.—F. R. H.

TEMPERATURE MEASUREMENT AND CONTROL

Status of Temperature Measurement in Steel Melting and Casting. K. Guthmann. (*Stahl u. Eisen*, 1955, **75**, Oct. 6, 1317–1322). The development of satisfactory immersion thermocouple and colour pyrometer equipments has given the operator facilities for temperature control in melting and casting. The value and usefulness of immersion and colour pyrometry in conjunction with radiation analysis are unmistakeable. The technical and economic importance in reducing rejects demands that they should be further extended and certain still doubtful relationships clarified.—J. P.

Temperature Measurement by Thermocouples: Theory and Practical Precautions. B. F. Billing. (*Eng. Insp.*, 1954, **18**, Autumn, 66–69, 76). The author describes the measurement of temperature by a thermocouple on commercially available instruments. The importance of a controlled cold junction is emphasized, and the principles of direct reading current-sensitive measuring instruments, potentiometric instruments, and broken thermocouple safety devices are described.

An Apparatus for Comparison of Thermocouples. T. M. Dauphinée. (*Canad. J. Phys.*, 1955, **33**, June, 275–285). A semi-automatic apparatus for comparing thermocouples of the same type in the temperature range 0–1100° C is described; the couples are welded together at the tips, and heated in a tube furnace. Comparisons of Pt/Pt–10% Rh couples may be made to accuracies within 0·3 μv (0·03° C) at a heating rate of 15° C/min, or 1·5 μv at 100° C/min. Provision is made for comparing corresponding elements of couples, for suppression of all or part of the measured e.m.f., and for measuring total e.m.f. when couples of different types are being compared.

REFRACTORY MATERIALS

Ceramics and Refractories. S. W. Sutcliffe. (*Soc. Chem. Ind., Reports on Progress of Appl. Chem.*, 1954, **39**, 44–56). Progress is reported on the following: clays and other raw materials, ceramic bodies, glazes and enamels, refractories, analytical and physical measurements and firing. During work on the dissociation of carbon monoxide by iron compounds, it has been found that an iron compound must be easily reducible to the metal, and that iron carbide is always an end product. The use of high-temperature cements consisting of mixtures of crushed aggregates and aluminous cement has been investigated. (75 references).—E. E. W.

The Orthosilicate–Iron Oxide Portion of the System CaO–"FeO"–SiO_2. W. C. Allen and R. B. Snow. (*J. Amer. Ceram. Soc.*, 1955, **38**, Aug., 264–280). The phase equilibrium diagram has been determined for the liquidus surface of the portion of the system, between fayalite, dicalcium silicate, wüstite and lime; detailed findings are given. Equilibria involving tricalcium silicate explain earlier observations on O.H. slags and furnace-bottom refractories.—D. L. C. P.

The System CaO–MgO–Al_2O_3–SiO_2. Phase-Equilibrium Relationships along the Join Anorthite–Spinel. J. H. Welch. (*J. Iron Steel Inst.*, 1956, **183**, July, 275–283). [This issue].

Relationships between Fundamental Ceramic Principles and the Wear of Basic Linings. N. Skalla and F. Trojer. (*Radex Rundschau*, 1955, July, 506–517). [In German]. Basic refractories are regarded as consisting almost entirely of crystalline phases whose texture and physico-chemical properties are the main factors controlling refractory behaviour and wear. Results are reported on various laboratory tests involving uniform and one-sided heating with and without external stresses. Effects of contamination, temperature changes, and insulation are also considered. Results are related to plant experience with O.H. linings.—E. C.

Refractory Manufacture. (*Metal Ind.*, 1955, **87**, Sept. 16, 237–240). A brief description is given of the plant, products, and methods of manufacture at The Derbyshire Silica Firebrick Co., Ltd. The main products are siliceous refractories made from the Derbyshire Pocket Silicas. Sand and clay preparation, mixing, and firing are dealt with.—P. M. C.

Production of Carbon Refractories from Petroleum Coke. H. P. S. Murthy and R. Singh. (*J. Sci. Indust. Res.*, 1955, **14B**, Nov., 597–602). Results are given of the preparation and properties of petroleum coke calcines, and an assessment has been made of the influence of quality and quantity of the binder.—E. E. W.

Improvement of Silica Bricks by the Addition of Aluminium Orthophosphate. G. Trömel and K.-H. Obst. (*Arch. Eisenhüttenwesen*, 1955, **26**, June, 307–318). The authors discuss the relationship between the crystal structures of silicon dioxide and aluminium orthophosphate, and the significance of the similarity of these structures with respect to the manufacture of silica bricks. Experiments in the manufacture of silica bricks with the addition of phosphoric acid or aluminium orthophosphate are described, and conclusions drawn from these experiments are given.—A. C.

Effect of Atmosphere on Refractories. H. M. Kramer (*Amer. Ceram. Soc. Bull.*, 1955, **34**, June, 173–176). This paper covers a number of factors of furnace operation which affect the life of refractories; blast- and O.H. furnaces are specifically mentioned. The factors include dusts and their absorption by the refractory, vapour pressure of certain materials normal to metallurgical processes, variation of percentages of gaseous components, and the effect of temperature alone on the constitution of important constituents of the refractory.—D. L. C. P.

Application of Photoelasticity to the Design of Tensile Specimens for Ceramic–Metal Materials. M. B. Stiefel. (*Amer. Ceram. Soc. Bull.*, 1955, **34**, May, 133–137). To evaluate various designs and grips used in the tensile testing of brittle materials, plastic models of several cermet specimens commonly in use were photoelastically investigated, and suitable designs selected for use.—D. L. C. P.

Refractories for Steelworks Ladles and Ladle Bricking Practices. C. W. J. Crawford and B. Wilcock. (*Iron Coal Trades Rev.*, 1955, **171**, Dec. 16, 1487–1491). A summary is given of the salient features of B.I.S.R.A.'s 45th Steelmaking Conference. The subjects included panel trials of various ladle bricks, use of fireclay bricks in ladles, lining construction, and the effect of chemical composition of the brick on slag–metal reactions in the ladle.—G. F.

Refractory Materials for the Vitreous-enamelling Industry. R. J. Slawson and J. F. Clements. (*Proc. Inst. Vitreous Enamellers*, 1952–53, 1953–54, **11**, 43–49, 52).

Effect of Intermittent Operation on Electric Furnace Refractories. R. P. Hill. (*Amer. Inst. Min. Met. Eng., Elec. Furn. Steel Conf. Proc.*, 1954, **12**, 87–88). An outline is given of the Sharon Steel Corporation's procedure in shutting down

electric furnaces and bringing idle furnaces back into production without damaging the refractories.—G. F.

Side Walls and Their Maintenance. R. J. McCurdy. (*Amer. Inst. Min. Met. Eng., Elec. Furn. Steel Conf. Proc.*, 1954, **12**, 67–69). The construction of electric-furnace side walls at South Chicago works of Republic Steel Corporation is described, and the maintenance procedure is outlined. Some mechanical and electrical equipment which helps to increase lining life is discussed.—G. F.

Evaluation of Performance of Electric-arc Furnace Refractories. M. P. Fedock. (*Amer. Inst. Min. Met. Eng., Elec. Furn. Steel Conf. Proc.*, 1954, **12**, 42–45). In comparing the effects of different melting practices on electric furnace refractories, the author evaluates refractory performance in terms of weight of brick consumed per ton of steel made. Results are presented for single-slag practice and for double-slag practice melting high and low carbon alloy steel, stainless steel, and low-alloy steel using oxygen.—G. F.

Dolomite as a Refractory Material for the Basic Air Refining Process. W. Bading. (*Stahl u. Eisen*, 1955, **75**, Oct. 6, 1300–1310). Because refractory consumption in the Bessemer process is much less than that in the open-hearth, the quality of converter linings has tended to be neglected. In order to rectify this position, a scheme has been set up whereby West European steelworkers can exchange experiences. The author discusses the effects of the sintering of dolomite, its chemical composition, and the burning of the raw material on its service behaviour, and describes the design of a modern dolomite plant. Converter bottoms are prepared by ramming, jolting, or vibrating and, if the appropriate machine is chosen, results by all three methods are comparable. The burning-in of bottoms, the composition of steelworks tar, the influence of particle size and packing density on bottom life are discussed.—J. P.

Beyond the Metals. V. Caldwell. (*Steel*, 1955, **137**, Aug. 1, 72–74; Aug. 8, 73–76). A review of super refractories, their properties and high temperature applications, is given. Oxides, carbides, nitrides, borides, and silicides are dealt with.—D. L. C. P.

Trends in German Silica Bricks for Open-Hearth Furnaces: Tests with Medium Conversion Material. K. Konopicky. (*Iron Coal Trades Rev.*, 1955, **171**, July 8, 102–104). Trials of German silica bricks in O.H. furnace roofs indicated that medium-conversion bricks give satisfactory service. Correlations with roof life show the desirability of low alumina content, and of low porosity and permeability.—G. F.

BLAST-FURNACE PRACTICE AND PRODUCTION OF PIG IRON

The Extraction and Refining of Metals. Edited by B. Fullman and F. D. Richardson. (*Soc. Chem. Ind., Reports on Progress of Appl. Chem.*, 1954, **39**, 129–161). The preparation of iron ore, blast-furnace practice, and the production of pig iron are reviewed, as well as steel melting and refining. The remarkable performance of the "Queen" blast furnaces at Appleby Frodingham is mentioned. (267 references).—E. E. W.

Single Blast-Furnace Operation in an Integrated Steelworks: Technique Practised without a Gasholder. J. W. Oliver and R. Kingston. (*Iron Coal Trades Rev.*, 1955, **171**, Sept. 2, 549–558). The integrated works of Skinningrove Iron Co., Ltd. is successfully operating a single blast-furnace system. The authors discuss the reasons for adopting the system, and give details of its operation and of the equipment and plant associated with it. Close liaison between departments is essential and the special signalling system adopted is described.

New Blast Furnace at Homecourt, France. (*Iron Coal Trades Rev.*, 1955, **170**, June 10, 1349–1350). Brief details are given of the design, main dimensions, top gear, and charging and ore handling systems of the new blast furnace installed at Homecourt. The furnace is of British design and French construction.—G. F.

The Design of Thin-Walled Blast-Furnaces. S. Černoch. (*Hutnické Listy*, 1955, **10**, (9), 513–521). [In Czech]. Czechoslovak experiences with the design and performance of thin-walled furnaces are analysed. The construction is considerably cheaper than that of conventional furnaces and the coke consumption about the same, but investment and maintenance costs are about half of those of conventional furnaces. Furnaces now being constructed in Czechoslovakia are of the thin-walled type, and existing thick-walled blast furnaces are being progressively converted to the former type. Full design details are given, including 23 drawings.—P. F.

Some Aspects of Blast-Furnace Operation. E. H. Baldwin. (*West Scotland Iron Steel Inst.*, Advance Copy, Paper No. 485, Dec. 16, 1955). The author points out that the operation of a blast furnace depends primarily on charging conditions, and then discusses in some detail the factors involved. It is stressed that it falls on the operator to watch the charge and to counteract variations, and the associated difficulties that arise because the furnace operates continuously and there is a time lag between observations and the effect of corrective action, are considered. The author also shows the use of operation records.

Tests on Elliptical Tuyeres. V. A. Sorokin *et al.* (*Stal'*, 1955, (2), 107–113). [In Russian]. After an account of previous experiments in the use of tuyeres of elliptical section, recent full-scale tests on an 18-ft hearth diameter blast furnace at the Stalin works are described. Details of the furnace operation with normal and elliptical tuyeres are given, and evidence is presented of increase in the active zone of the hearth with the latter. Best effects were obtained when ores were charged preferentially to the walls. With elliptical tuyeres the pressure-drop up the furnace decreased, enabling higher blowing rates and blast temperatures to be used, the productivity increasing by 3·03%, and coke rate decreasing by 2·84%. The lives of the two types of tuyeres were equal.

Sintered Ore—New Glow in Blast Furnace Economy. T. F. Hruby and R. M. Love. (*Steel*, 1955, **137**, Sept. 26, 112–114). Past, present, and future employment of sinter in blast-furnace burdening is considered. The increasing use of sinter may double U.S. capacity in the next 10 years.—D. L. C. P.

Ore Fines Utilized in Low Shaft Furnace to Produce Thomas Pig Iron. P. Coheur. (*J. Met.*, 1955, **7**, Aug., 872–876). The experimental low-shaft furnace in Belgium has successfully produced low-silicon iron from materials, including ore fines, which could not be used in the conventional blast furnace. The pilot plant is described, test conditions are outlined, and operating data are given. The general conclusions from the trials are listed.—G. F.

Desulphurization of Liquid Pig Iron by Blowing with Lime Powder. B. Trentini, L. Wahl, and M. Allard. (*J. Iron Steel Inst.*, 1956, **183**, June, 124–133).

Desulphurizing Power of Acid Blast-Furnace Slags. V. I. Loginov. (*Stal'*, 1955, (2), 119–127). [In Russian]. An account is given of an investigation, by laboratory experiments and by study of works data, of desulphurization by slags, with special reference to acid slags. In the laboratory experiments, multiple-compartment graphite containers were used for the reactions, one of the compartments containing a control slag. The partition of sulphur between slag and metal in relation to slag basicity, composition, and temperature was determined.—S. K.

The Chemical Behavior of Silicon in the Iron Blast Furnace. J. C. Fulton. (*Reg. Tech. Meet. Amer. Iron Steel Inst.*, 1954, 113–126). The author reviews recent investigations contributing to knowledge of the behaviour of silicon in the blast furnace, and particularly in the high-temperature reactions in the hearth. The relation between silicon and sulphur is discussed, and the effects of desulphurization with acid and basic slags are considered. Finally, research on the silicon equilibrium, and the manner in which sulphur influences it, is described.—G. F.

Solid State Diffusion in the Reduction of Magnetite. J. O. Edstrom and G. Bitsianes. (*Trans. Amer. Inst. Min. Met. Eng.*, 1955, **203**; *J. Met.*, 1955, **7**, June, 760–765). The authors have determined the parabolic rate constants for the formation of wüstite by the diffusion-controlled solid-state reaction between magnetite and iron. Inert marker studies show that mass transport through the wüstite layer is accomplished by iron migration. The transport capacity for iron through dense wüstite layers is found to be sufficient to carry on reduction, even in gaseous reduction processes. Relationships between rate constants and self-diffusivities are discussed.—G. F.

Machine Casting of Electrically Smelted Iron. Two-Strand Machine for Italy. (*Engineering*, 1955, **180**, Aug. 5, 183). This article describes a machine designed and built by Sheppard & Sons Ltd., Bridgend, Glam. for Nazionale Cogne to deal primarily with the iron output from three Lubotti 3500 kVA electric smelting furnaces. The extreme fluidity

of the very high temperature electrically-produced iron presents special difficulties.—M. D. J. B.

TREATMENT AND USES OF SLAG

Slag Cement and its Use as Low Heat Cement. C. H. Khadilkar. (*J. Sci. Indust. Res.*, 1955, **14A**, Aug., 385–388). Investigations are being made with a view to producing low-heat cement in India. A review of methods is given. It is hoped that all the cement required for river valley projects will be provided by the existing cement and iron works and those to be established in the near future.—E. E. W.

Processing of Road-Making Slags at the August Thyssen Steelworks. E. Grosse-Weischede. (*Silicates Indust.*, 1955, **20**, Dec., 470–476). [In German]. Processing of blast-furnace slags in this Ruhr steelworks is described.—P. F.

The Production of Hydraulic Cements from Blast-Furnace Slags Ground With Small Quantities of Chalk and Sodium Salts. A. O. Purdon. (*Silicates Indust.*, 1955, **20**, Nov., 424–427).

Blast-Furnace Cements in Italy. G. Malquori. (*Silicates Indust.*, 1955, **20**, Oct., 377–379). [In Italian]. The production and applications of slag cements are described.—P. F.

PRODUCTION OF STEEL

Youngstown in Chicago. T. J. Ess. (*Iron Steel Eng.*, 1955, **32**, Sept., 71–726). This article gives a detailed description of all the departments and activities of the 5½ million ingot ton works of the Youngstown Sheet and Tube Co. The raw materials, supplies, coke ovens, blast furnaces, steel plant, and numerous rolling mills are described as well as the operating conditions, production capacities, maintenance schemes, and services.—M. D. J. B.

British Furnace Developments. D. Llewellyn. (*Metal Ind.*, 1955, **86**, Apr. 29, 339–344). Modern trends in melting and heat-treatment furnace design are discussed, and the author gives descriptions and illustrations of some recently installed plant, including oil-fired rotary furnaces and large electric-arc furnaces.—P. M. C.

Influence of Raw Materials, Power Supplies, and Economic Factors upon the Choice of Steel Making Method. E. Krebs and K. H. Schnittger. (*Inst. Hierro Acero, Special Number*, 1955, **8**, Oct., 475–495). [In Spanish]. A critical discussion of these factors in relation to open-hearth, oxygen-blowing, and electric-arc steelmaking practices is given. (35 references).

Special Problems of Blast Supply in Steel Works. H. Pötzschner and J. Tischendorf. (*Metallurgie*, 1955, **5**, Sept., 272–280). Methods of assessing blast requirements in steel-works are described; practical examples are given; suggestions for the choice and layout of blowing plant are made.—L. J. L.

The New Works of "Forges et Aciéries de Dilling." (*Usine Nouvelle*, 1955, **11**, Sept. 22, 59–61). Plant capacities at the Dilling works are summarized. The overall steel production is 50,000 tons/month. Details are given of the modernized and rebuilt large sheet-iron works, sheet finishing equipment, and the tinning plant.—T. E. D.

Iron and Steel in Canada: An Integrated Industry West of the Canadian Rockies. G. P. Contractor. (*Iron Steel*, 1955, **28**, July, 347–351; Aug., 384–386). The author discusses the reserves and production of iron ore and coal, and the production and consumption of steel and pig iron, in British Columbia. He concludes that the possibility of an integrated industry in the area depends on a sufficient tonnage of iron ores being found.—G. F.

Manufacture of Iron and Steel. G. Bulle. (*Tidsskr. Kjemi, Bergvesen Met.*, 1955, **15**, (3), 37–44). [In German]. A review of problems associated with the metallurgy of scrap, ore, and crude iron deals with the shortcomings of the O.H. furnace, advantages of the oxygen-blast and hot-blast cupola, comparison between high- and low-shaft furnaces, and possibilities of process rationalization, waste-heat utilization, and the increasing use of the oxygen converter. Main operating data are given for eight furnaces illustrated.—G. G. K.

Some Recent Engineering Developments in the Iron and Steel Industry. J. A. Kilby. (*Proc. Staff. Iron Steel Inst.*, 1946–53, **61**, 95–116). A brief account of modern equipment in steelworks is given. The design of cranes, O.H. furnaces, soaking pits, and rolling mills is considered.—B. G. B.

Five Years of the Steel Research Institute at Henningsdorf near Berlin. E. Maurer. (*Technik*, 1955, **10**, Aug., 449–456). The central steel research institute of the East German Republic and some of the investigations carried out since its formation in 1950 are described; these are exclusively *ad hoc* investigations of metallurgical problems arising in East German steelworks.—J. G. W.

Pneumatic Steelmaking Processes. D. J. Carney. (*Blast Furn. Steel Plant*, 1955, **43**, June, 635–640; July, 753–760; Sept., 1006–1010; Oct., 1139–1141). Pneumatic steelmaking processes in use today are reviewed. Acid and basic steel-making using bottom, side, and top blowing methods are considered. The design of converters, types of pig iron used, stages in the making of commercial heats, the chemistry of the processes, and control of the end point, temperature, and nitrogen content are examined. The advantages to be gained by the large-scale use of oxygen are discussed.—B. G. B.

Trends Revealed by the Study of the Transformation of Thomas BTM Steel Made using Oxygen Enriched Air. P. Leroy and J. Stremsdoerfer. (*Publ. Inst. Rech. Sid.*, Series A, 1955, July, (105), 33). Thomas steels, low in nitrogen, phosphorus, and sulphur, produced by a method developed by IRSID, were submitted to rolling and wire-drawing trials at various works. Detailed results are given, including changes in mechanical properties, segregation data, and photomicrographs. It is concluded that these preliminary results indicate a definite improvement over normal Thomas steel.—T. E. D.

Control of the Bessemer Converter Reaction: Use of Radiation Pyrometry and Flame Spectroscopy. (*Iron Coal Trades Rev.*, 1955, **170**, June 17, 1413–1414). This article summarizes recent German work in which radiation pyrometry and flame spectroscopy were studied as means of indicating the stage of the Bessemer converter reaction. It is concluded that, if both techniques are functioning correctly, the degree of control would approach that of the slower-working O.H. furnace.—G. F.

Control in the Acid Bessemer Process. Application of Volume Debitgraphe, Opacimeter, and Flame Pyrometer. P. J. Leroy, J. G. Galey, and F. B. Cawley. (*J. Iron Steel Inst.*, 1956, **183**, June, 208–224).

French Metallurgical Developments: Autumn Meeting of the Société Française de Métallurgie. T. Bishop. (*Iron Coal Trades Rev.*, 1955, **171**, Nov. 11, 1165–1168). Following a survey of specific problems of the French iron and steel industry, summaries are given of some of the papers presented at the Autumn Meeting of the Société Française de Métallurgie. Particular attention is given to the blowing of basic Bessemer steel with a mixture of oxygen and carbon dioxide.—G. F.

A Wide-Field High-Temperature Periscope. C. Burns. (*Trans. Soc. Instrument Techn.*, 1955, **7**, Dec., 137–146). The design, construction, and mode of operation of the B.I.S.R.A. furnace-scanning periscope are described, in its application to O.H. visual and photographic observations. The field of view is 47½°, and a modification is described which reduces the field to 40° but gives less risk of splashing. Photographic techniques using the instrument are described.—L. D. H.

Report of the "Furnace-Construction" Sub-Committee. (*Centre Doc. Sidér., Circ. Inform. Tech.*, 1955, **12**, (10), 1980–1982). The construction of front walls of O.H. furnaces is briefly discussed and the design of skewbacks is mentioned. A water-cooled skewback with basic brick protection and with thicker roof bricks near the furnace wall, and also a double skewback are shown. The construction of slag pockets and problems related to them are discussed.—T. E. D.

Study on a Model of Aerodynamics of a Maerz-type Open-hearth Furnace with Preheated Gas and a Comparison with Siemens and Terni Furnaces. G. Husson, G. Cohen de Lara, and R. Durand. (*J. Iron Steel Inst.*, 1956, **183**, June, 134–145).

Open-Hearth Steel from Basic Bessemer Iron Rich in Phosphorus. W. Oelsen and H. Voigt. (*Stahl u. Eisen*, 1955, **75**, Aug. 11, 1013–1024). The reasons for wishing to produce O.H. steel from high phosphorus, silicon-containing iron from Salzgitter ores are discussed, and the development of a suitable method is described. By careful attention to the charging process and to the working of the heat, which proceeds in three stages, it has been possible to effect dephosphorization before the removal of carbon. The good desulphurization of the steel makes the process economically useful under conditions of poor raw materials. The slag is essentially similar to that from the basic Bessemer and can be employed as a high-grade fertilizer. The amount of iron lost in the slag is a little less than in the Bessemer process.—J. P.

Shortening Standstill Periods Due to Open Hearth Furnace Repairs by Blasting the Slag. A. S. Akantisz. (*Maschinenwelt Elekrotechnik*, 1955, **10**, July, 175–176). A substantial saving

of time in clearing the slag chamber is claimed by the use of the " slagjack " by means of which the slag chamber can be drilled before the furnace is taken off.—L. D. H.

Ore or Mill Scale as Decarburizing Agent in the Steel Furnace. E. Plettinger. (*Radex Rundschau*, 1955, May/June, 409–417). [In German]. The decarburizing process is discussed and the respective merits of using manganese-bearing ores or mill scale are considered in detail. The use of ores is regarded as preferable because of higher purity, greater convenience, saving in costs by obviating manganese additions, and incidental benefits in connection with desulphurization.—E. C.

Contribution to the Pig-Iron/Ore Process Technology. A. Děkanovský. (*Hutnické Listy*, 1955, **10**, (7), 403–408). [In Czech]. Works tests on large stationary and tilting O.H. furnaces showed that output increased with increasing pig-iron temperature in the charge, and decreased with increasing content of carbon, silicon, and manganese in the iron.—P. F.

Desulphurization of Steel in the Open-Hearth Process: Relation Between Carbon Content and Slag Composition. E. T. Turkdogan. (*Iron Coal Trades Rev.*, 1955, **171**, Dec. 16, 1471–1476, 1478). From a study of works' data, the author shows that carbon content of the bath can be related to slag composition, which enables the oxidizing power of O.H. slags to be evaluated. The data show a reasonably good relationship between the sulphur capacity (S)/[S](ΣFe) and total acids ($SiO_2 + P_2O_5$) of the slag in the basic O.H. process, and this simple relationship might be used as the basis for constructing a method of slag control for sulphur removal.—G. F.

Conditions for the Preliminary Deoxidation of the O.H. Baths. P. G. Glazkov. (*Stal'*, 1955, (2), 128–132). [In Russian]. An investigation is described of the influence of the technique used for deoxidizing the O.H. baths with blast-furnace ferro-silicon and ferromanganese on the deoxidation process and the quality of the steel. Preliminary deoxidation with 12% ferrosilicon does not improve the quality of low-carbon metal and ferromanganese alone should be used.—S. K.

An Investigation on the Function of Ingot Mould Dressings. D. R. Thornton. (*J. Iron Steel Inst.*, 1956, **183**, July, 300–315). [This issue].

The Casting of Forging Ingots in Vacuo. A. J. Zuithoff. (*Metalen*, 1955, **10**, Dec. 15, 498–499). [In Dutch]. The article briefly describes a method developed by the Bochumer Verein in Germany for the casting of large forging ingots in vacuo with the object of releasing harmful gases, notably hydrogen.—F. R. H.

Influence of Ingot Shape and Teeming Time upon Ingot Surface Defects. A. Herrero. (*Inst. Hierro Acero*, 1955, **8**, July-Sept., 365–369). [In Spanish]. A study has been made of 1991 basic electric steel, octagonal ingots of from 3·1 to 60·5 tons by classifying the surface defects into four types and giving each a weighted value according to its seriousness. This index, when plotted against $h/\sqrt{Sm}$, where h = ingot height, Sm = average section, gave a minimum about the value $h/\sqrt{Sm} = 4$. The same index was plotted against the teeming rate for 144, 11·1-ton ingots cast with a 28 mm nozzle and gave a minimum at 8·5 to 9·5 min. It is also pointed out that close temperature control is necessary, and that different casting techniques must be employed according to size and shape of ingot.—P. S.

The Nature of Mottled Segregation. A. M. Ofengenden, Ya. L. Troskonov, and L. M. Pokrass. (*Stal'*, 1955, (2), 152–158). [In Russian]. This article deals with the nature of mottled segregation and of the effects on it of various factors, with special reference to low-carbon, low-alloy silicon steel. Mottled segregation in transverse sections of rolled metal is attributed to local accumulation of segregations, mainly at and near the central zone, produced by the movement of gas bubbles up the ingot. In ingots of low-carbon steel alloyed with silicon, mottled segregation is connected with the evolution and upward movement during solidification of dissolved hydrogen. In low-alloy chromium–nickel–silicon steel, mottled segregation and similar defects are due to the high hydrogen content of the 75% ferrosilicon used for introducing the silicon; they can be avoided by preliminary heating of the ferro-alloy to 1100–1150° C in coal-fired furnaces for 8 h. Other causes to which high gas content of steel is attributed are: excessive charging of gas-saturated steel, excessive use of lime and bauxite in slag, low slag basicity during melting, insufficient boiling of the bath. No benefit is obtained by the use of strong deoxidizers if this is associated with increased hydrogen content of the steel. Mottled segregation does not always lower the mechanical and technological properties of steel measured with longitudinal test-pieces; hence it is less serious in rolled products. In high-carbon steels the defect is due to oxidation of silicon in the ladle through insufficient deoxidation; the silicon content of the finished steel should be $\geq 0{\cdot}22\%$.—S. K.

PRODUCTION OF FERRO-ALLOYS

Separation of Tantalum and Niobium. (*Tek. Tidskr.*, 1955, **85**, Sept. 13, 739–742). [In Swedish]. A review of the most promising separation methods briefly describes results obtained and procedures employed in liquid–liquid extraction, the Belgian Geomines method of continuous counter-current extraction from low-grade, high iron content ores, and selective chlorination. The production of metallic tantalum is also touched upon.—G. G. K.

Electrolytic Production of Manganese from Low Grade Ores. A. Jogarao, Y. D. Prasada Rao, and S. Gopal. (*J. Sci. Indust. Res.*, 1955, **14B**, Sept., 467–472). A daily output of 10 lb of 99·9%-purity manganese has been obtained in a semi-pilot plant using low-grade manganese ores (about 30% Mn) by an electrolytic process, details of which are described.—E. E. W.

FOUNDRY PRACTICE

The Control of Castings in the Foundry. G. Storek. (*Giesserei*, 1955, **42**, Sept. 29, 567–568). A short article in which the control and instructions necessary for different types of castings during their passage through the foundry are followed. Inspection of the castings to ensure that they have reached the required acceptance standards is discussed. The usual controls, at present applied in the foundry, and their failings are described.—R. J. W.

Environment in the Foundry. (*Mod. Castings Amer. Foundryman*, 1955, **28**, July, 47–78). The problems of improving working conditions in the foundry are considered under the headings hygiene, ventilation, hoods and systems, sand handling, mould and core making, melting and pouring, radiation, cleaning rooms, welding and woodworking, dust and fume collection, maintenance and testing, and housekeeping.—B. C. W.

Improving Foundry Paperwork. M. E. Mundel. (*Amer. Found. Soc., Preprint*, No. 142, 1955). Some improvements that may be made in the internal paperwork of a foundry are briefly discussed.—B. C. W.

What Design Engineers Look for in Castings. T. Davidson. (*Amer. Foundryman*, 1955, **27**, May, 118–122). The points which are considered by design engineers in deciding the suitability or otherwise of castings for a particular application are discussed and illustrated by reference to a number of practical examples.—B. C. W.

General Motors' Experimental Foundry. L. J. Pedicini. (*Amer. Foundryman*, 1955, **27**, May, 110–117). A description is given of the experimental foundry established at the General Motors Technical Centre near Detroit. The facilities provide for both ferrous and non-ferrous melting and casting, and include a production pilot line and shell-moulding equipment.—B. C. W.

Mechanized Foundry for Cast Iron Parts. B. Ya. Kompaniets. (*Liteĭnoe Proizvodstvo*, 1955, (4), 8–10). [In Russian]. Two designs of mechanized foundry for the production of up to 1000 tons of cast-iron utensils per year are described. In the first, the whole output is from chill moulds; in the second, chill moulds are used for only a small proportion, the bulk being produced in earth moulds. Comparison of the respective productivities, outputs, material consumptions, labour requirements, costs, and capital costs show the superiority of the chill-mould design except where flexibility is likely to be the over-riding requirement.—S. K.

Minor Mechanization in Repair-Foundry Works. S. E. Utkin and V. A. Nazaretskiĭ. (*Liteĭnoe Proizvodstvo*, 1955, (5), 24–26). [In Russian]. Some examples are given of possible mechanization of operations in the complex conditions of repair-foundry work, including sand-drying, cupola-charging, and core-drying with quick-drying mixtures.—S. K.

Metallurgy of Cast Iron H. O. Reisener. (*Engineer Foundryman*, 1955, **20**, May, 65–67; June, 52–59). An elementary account of the production, foundry practice, and metallography of cast iron.—A. D. H.

Hot Blast Cupolas: A Review of Types at present Operating in Great Britain. G. J. Shaw. (*Iron Steel*, 1955, **28**, Sept., 415–420). The author summarizes the advantages to be

obtained in hot-blast cupola operation, and discusses systems operating in Great Britain using independent blast preheat, preheat by combination of cupola gas, and preheat by direct heat transfer from cupola gas. Details of operating techniques and performance are included.—G. F.

The Melting of Chips in Cupolas by Croft's Method. S. H. Chrobok. (*Giesserei*, 1955, **42**, Aug. 4, 409–412). The methods of introducing chips into both hot- and cold-blast cupolas are first described and illustrated. Works results indicate that Croft's method is suitable for both types of furnace. A study of the economics of this method shows that considerable savings may be effected using cast-iron chips. Mechanical charging methods are described. Testing shows that the burning loss, oxidation, and sulphur contents do not exceed normal values and, further, that it is possible to melt steel and malleable cast-iron chips by this method. The analysis and properties of the irons were in no way altered.

Melting Practice in Iron Casting. G. Speer. (*Giessereitechn.*, 1955, **1**, Sept., 166–168). The author discusses the cupola, refractories, melting processes, the hot-blast cupola, and the electric furnace.—L. J. L.

Determination of the Actual Amount of Air Introduced into a Cupola. A. Dahlmann. (*Giesserei*, 1955, **42**, Aug. 18, 440–442). The ideal gas equation is first used to calculate the amount of air blast required under normal conditions. Numerical examples are given. The derivation of formulæ for calculating the amount of air used in the furnace is described. Graphical examples of their uses are given. Calculations of the amounts of air lost can be made.—R. J. W.

Experiments in a Basic-Lined Cupola with Hot-Blast and Coke Encrustation. M. G. Frohberg and W. Döbber. (*Giesserei*, 1955, **42**, Oct. 13, 580–585). A series of melting experiments have been carried out in a small cupola furnace which showed that proposals for coke encrustation with Portland cement (by E. Piwowarsky and H. Eyckeler) were not satisfactory. Experimental results are given for iron temperature, iron analysis, throat-gas behaviour, and the mechanical properties of the irons used. The experimental method used is clearly described.—R. J. W.

Try Gray Irons for High Temperature Service. J. L. Everhart. (*Mat. Methods*, 1955, **41**, Feb., 90–93). The many satisfactory applications of grey cast irons, particularly the alloyed compositions, for high-temperature service are reviewed. Alloyed irons may be obtained with tensile strengths up to 50,000 lb/in², and are used extensively for automotive and diesel cylinder blocks, liners and heads, steam-control valves, furnace liners, stokers and grates, and many other applications.—P. M. C.

Thermal Shock. Some Experiments with Cast Iron. E. R. Evans. (*Brit. C.I. Res. Assoc. J. Res. Dev.*, 1955, **5**, June, 643–654). An apparatus to heat and quench cast-iron specimens automatically and repeatedly according to a predetermined cycle is described. The effect of variations in composition on the thermal shock resistance of cast iron has been studied.—B. G. B.

Continuous Casting of Grey Iron. A. N. Myasoedov and I. R. Dudnik. (*Liteinoe Proizvodstvo*, 1952, **3**, (11), 2–5). [In Russian]. A description of the operating principles and the design of the continuous-casting machine are given. Quality of continuously cast grey iron with that cast in sand and chill moulds is compared. The relationship between microstructure, cross-section of the billet, and cooling rate is discussed.—V. G.

Construction of an Experimental Graphite Resistance Furnace. E. A. Chard. (*Iron Coal Trades Rev.*, 1955, **171**, Aug. 26, 495–497). A description is given of the design and construction of an experimental furnace, heated by a graphite resistance unit and used for rapid heating of small slabs to 1100° C. The furnace reaches temperature in about 3 h and the slab can be heated to 1100° C in about 15 min.—G. F.

Removal of Lead from Cast Iron in the Induction Furnace. G. Östberg. (*Gjuteriet*, 1955, **45**, July, 96–98). [In Swedish]. Laboratory tests with 2-kg melts carried out to determine optimum conditions for Pb removal in induction furnaces by evaporation at constant temperatures showed that best results were obtained at 1350° C. The process did not seem to be impeded by a thin slag blanket, whilst bubbling through He did not affect the rate of removal. The experiments also gave a qualitative measure of the solubility of Pb in cast iron containing 3% C and 2% Si, this being: at 1300°, 0·020%; at 1350°, 0·063%; and at 1450°, 0·110% Pb.—G. G. K.

The Role of Surface in the Graphitization of White Iron. K. P. Bunin, Ya. V. Grechnyĭ, and N. M. Danil'chenko. (*Liteĭnoe Proizvodstvo*, 1955, (5), 12–15). [In Russian]. This is a study of the graphitization of white iron with special reference to the role of internal and external surfaces. An important part in graphitization is assigned to the diffusion of vacant lattice spaces, which is affected by surface conditions.—S. K.

Investigation of the Mechanical Properties of White Iron and the Stresses in It. L. I. Kozhinskiĭ. (*Liteĭnoe Proizvodstvo*, 1955, (4), 19–22). [In Russian]. The investigation described is a first stage in the study of the nature of cold cracks in malleable iron castings, and involved the determination, using special test pieces, of the mechanical properties, structures, and casting stresses of white irons of different compositions. The effects of C, P, S, Mn, and Si contents of the cast metal have been investigated.—S. K.

Calcium Carbide Desulphurisation By the Injection Process. J. A. DeHuff and R. Schneidewind. (*Amer. Found. Soc., Preprint*, No. 26, 1955). The mechanism of desulphurization in the calcium carbide injection process is briefly considered, together with the factors affecting the efficiency of the method. Experimental results obtained with single- and multiple-stage injection are presented and discussed. The irons treated by injection were found to show an increase in matrix ferrite and a trend to finer graphite with decreasing S contents.

Increasing Carbon Content of Cast Irons By Ladle Injection. G. E. Spangler and R. Schneidewind. (*Amer. Found. Soc., Preprint*, No. 27, 1955). Results obtained in the laboratory and foundry show that the C content of molten iron can be substantially and uniformly increased by injecting a suitable form of graphite using dry N_2 as a carrier. Desulphurization may be achieved at the same time by injecting calcium carbide with the carbon. Carbon injected irons have similar properties to normal grey irons of the same composition.—B. C. W.

New High Strength Cast Irons Produced By Injection Methods. J. W. Estes and R. Schneidewind. (*Amer. Found. Soc., Preprint*, No. 28, 1955). The development of a class of high-strength cast irons intermediate in properties between normal grey cast iron and nodular iron is described, with details of the range of mechanical properties. They are produced by injecting a mixture of calcium carbide and rare-earth oxides with or without MgO; 20–40% of the graphite is nodular, the remainder being present as short stubby flakes. The advantages of the new 'upgraded' irons are their high strength, ductility, insensitivity to changes in section size, low chill depth, and low solidification shrinkage. To upgrade cold metal it is necessary to inject Mg metal with the calcium carbide, and this technique may also be used to produce a fully nodular iron. The advantages of producing nodular irons by the latter method are summarized.—B. C. W.

Nickel Austenitic Ductile Irons. F. G. Sefing. (*Amer. Found. Soc., Preprint*, No. 32, 1955). The properties of three typical Mg-treated Ni austenitic cast irons are reviewed and compared with those of a similar iron with flake graphite. The properties considered include normal mechanical and physical properties, elevated temperature properties, and corrosion resistance. The manufacture, heat-treatment and industrial application of the Ni austenitic nodular irons are briefly discussed.

Applications of Spheroidal Graphite Cast Iron at High Temperatures—Resistance to Oxidation and Creep Strength. C. Galletto. (*Ing. Mecc.*, 1955, **4**, June, 25–33). [In Italian]. This paper discusses two important, if little known, properties of S.G. iron which make it possible to use this material in place of much more expensive metals. These are the resistance to oxidation and expansion and the creep strength of p ain high-Si and high-Ni austenitic S.G. irons. The author shows that these have a higher resistance to oxidation than grey cast iron, comparable to that of 12% Cr stainless steel. As far as creep is concerned, at temperatures of 425–650° C, plain S.G. iron can be compared to C and to certain Cr–Mo steels. The use of S.G. iron can be recommended for long-term load-carrying applications at temperatures of 540–595° C when both resistance to spalling and creep are required. Austenitic S.G. irons can be used up to temperatures of 705° C and even higher when loading is not excessive.

Experiments on the Inoculation of Cast Iron with Magnesium. S. I. Vitenzon, R. S. Tripol'skaya, and R. I. Galaiko. (*Liteĭnoe Proizvodstvo*, 1955, (5), 18–19). [In Russian]. The skulls remaining in the ladle after inoculation of a cast iron with Mg, and with Mg plus FeSi, were examined with special reference to heterogeneity.—S. K.

Investigation of the Growth Stability of Magnesium Iron. A. D. Ushakov. (*Liteĭnoe Proizvodstvo*, 1955, (4), 22–23). [In Russian]. In the investigation described, dilatometric curves were obtained for a Mg cast iron with nodular graphite. Specimens in the pearlitic and ferritic states were heated at 3–4° C/min to 500°, 700°, and 800° C, being soaked at these temperatures for 3 h. Parallel determinations of mean coefficients of linear expansion gave values for the Mg iron 6–7% higher than those for ordinary grey iron with lamellar graphite. The differences observed in growth stability are discussed in terms of structure and oxidation.—S. K.

Malleable Duplexing Melting Control. F. L. Lopour. (*Amer. Foundryman*, 1955, **27**, Apr., 38–39). The methods used at the Belle City Malleable Iron Co., Racine, Wis., to control the quality of cupola iron being supplied to a reverberatory furnace are briefly described.—B. C. W.

Start Duplex Malleable Control with Raw Materials. L. E. Emery. (*Amer. Foundryman*, 1955, **27**, June, 90–95). The methods used at the Marion Malleable Iron Works Division of the Chicago Railway Equip. Co., Marion, Ind., for controlling the production of duplexed malleable iron are described and discussed.—B. C. W.

Intensification of the Annealing of Malleable Iron. I. I. Gurdus and K. I. Kazakevich. (*Liteĭnoe Proizvodstvo*, 1955, (5), 26–27). [In Russian]. The rapid annealing of malleable iron based on a preliminary soaking at about 750° C is described.—S. K.

Modern Methods of Production of Malleable Iron. J. Piaskowski and J. Raczka. (*Przeglad Odlewnictwa*, 1955, **5**, (6), 161–168). [In Polish]. Principles of choosing the type of furnace for melting malleable iron depending on its quality and the organization of the production in a foundry are outlined. Melting furnaces for continuous and intermittent production, their output, melting practices used, and the chemical composition and properties of metal produced are discussed.—V. G.

The Effect of Mould Material on Mould Capacity and Heat Crack Formation in Steel Castings. F.-K. Althof and R. Radtke. (*Giessereitechn.*, 1955, **1**, July, 111–115). Practical experience has shown that the material of which a mould is made tends to inhibit shrinking, and that the composition of this material also plays a part. The authors first discuss mould capacity, experimental apparatus, steel composition, and heat cracks. In the second part the authors deal with various methods of determining tendency to heat crack formation. Tests have shown that the binding agents exert a considerable influence.—L. J. L.

Effect of the Form of Pearlite and the Ferrite Grain Size on the Properties of Steel Castings. M. M. Kantor and N. M. Serpik. (*Liteĭnoe Proizvodstvo*, 1955, (5), 19–21). [In Russian]. An investigation is described of the effect of the form of the structural components of annealed steel castings on various mechanical-property parameters. Specimens were subjected to various heat-treatments giving lamellar or granular pearlite. The tensile properties were determined and related to the form of the pearlite.—S. K.

High-temperature Resistant Alloy-steel Castings. (*Usine Nouvelle*, 1955, **11**, Sept. 1, 53–57). The properties of alloy-steel castings for service above 600° C are outlined. The steel-making methods and casting techniques are described. The choice of high-temperature steels for particular purposes is explained.—T. E. D.

Progress in the Production and Use of High-alloy Steel Castings for the Chemical Industry. F. Pölzguter. (*Werkstoffe Korrosion*, 1955, **6**, Aug.-Sept., 375–385).

High Temperature Properties of Sands Containing Coal Dust and Woodflour. R. G. Godding. (*Brit. C.I. Res. Assoc. J. Res. Dev.*, 1955, **6**, Aug., 30–35). The relationship between the strength and temperature of synthetic sand is shown for two different rates of heating. Curves showing the relationship between temperature and strength of a plain clay-bonded sand and deformation and brittleness of fracture are reproduced. The effect on these properties of additions of coal dust are given.—B. G. B.

Bonding Characteristics of Fulbonds and Scope for Development of Indigenous Fullers' Earth. R. M. Krishnan, M. N. Khanna, and B. R. Nijhawan. (*J. Sci. Indust. Res.*, 1955, **14A**, Nov., 531–534). During the war, the production of steel castings by green sand practice gained considerable impetus in the U.K. Tests are described on various clays to find a suitable indigenous bonding material to replace Wyoming bentonite.—E. E. W.

I. The Investigation of Cement Sand. II & III. Cement Sand as a Moulding Material. H. G. Levelink. (*Metaalinstituut TNO*, Publ. 30, 1955, Jan., 1–7; 8–12; 13–16). [In Dutch]. The first paper concerns a study of the behaviour under various conditions of cement moulding sands resulting in the development of a routine laboratory testing method. Comparison is also made of certain brands of cement on the market. The second paper is a summary of the results obtained from an enquiry among the Dutch foundries. The third paper covers points requiring consideration when adopting the cement–sand moulding method, notably the materials and their preparation, transport within the foundry, the moulding procedure, core properties, equipment, and the necessary changeover.—F. R. H.

How to Measure Moulding Sand Blowability. D. M. Murray. (*Amer. Foundryman*, 1955, **27**, June, 78–81). A method of estimating the ease with which a sand can be blown into a mould is described and the results of some tests on two sands with varying blowing pressures and moisture contents are reported.—B. C. W.

Casting Precision Patterns in Zircon Moulds. J. E. Stock and A. V. Schoville. (*Amer. Foundryman*, 1955, **27**, Apr., 34–37). A method is described for producing cast iron patterns to close tolerances using a zircon facing sand. The cost of producing the cast-iron pattern is comparable with that for any other jobbing casting but considerable time is saved in finishing.—B. C. W.

Mixtures for the Thermal Insulation of Risers on Castings. R. Kamenský and A. Kubacký. (*Slévárenství*, 1955, **3**, (11), 356–359). [In Czech].

Contribution to the Theory and Practice of Atmospheric Risering. M. Houšt. (*Slévárenství*, 1955, **3**, (10), 322–324). [In Czech]. The principles of correct usage of risers are discussed, and experimental verification of the theoretical conclusions is adduced.—P. F.

Risering Ductile Cast Iron. R. A. Flinn, D. J. Reese, and W. A. Spindler. (*Amer. Foundryman*, 1955, **27**, Apr., 62–66). Data to show the effects of side-blind riser size and position on the soundness of bar and plate castings in ductile iron are presented and discussed. A curve is given from which the minimum riser size to avoid riser-neck shrinkage in a given casting may be determined. The results obtained for the effective feeding distance of risers are in good agreement with those of Pellini on cast steel.—B. C. W.

Core Room Pitfalls. R. H. Greenlee. (*Mod. Castings Amer. Foundryman*, 1955, **28**, Sept., 36–38). The factors which must be carefully controlled to produce satisfactory cores at a low cost and with the minimum scrap are briefly discussed.

New Sugar Formula Makes Sweet Core Binder. C. J. Gogek. (*Mod. Castings Amer. Foundryman*, 1955, **28**, Aug., 55–57). Results of laboratory tests are presented to show that corn sugar with 10% ammonium sulphate as catalyst may be used as a satisfactory core binder for green sand.—B. C. W.

The Production of Sand Cores and Moulds by Means of the CO_2 Process. R. C. Cross. (*Engineer Foundryman*, 1955, **19**, Apr., 56–64). The advantages and limitations of the CO_2 process in which sodium silicate binder is decomposed to form a silica gel bond by contact with CO_2 gas is described. Properties of suitable sands and methods for the introduction of the gas are discussed.—A. D. H.

How to Bake Cores Without Heat. W. Schumacher. (*Mod. Castings Amer. Foundryman*, 1955, **28**, July, 32–35). The use of the CO_2 process for hardening moulds and cores is briefly described and examples of the successful application of the method at the German steel foundry of Concordiahütte, Bendorf am Rhine are illustrated and discussed. The advantages of the process over the normal method of oven drying are summarized.—B. C. W.

Time Studies in the Hand Molding Shop. W. Bernard. (*Giesserei*, 1955, **42**, July 21, 392–397). The division of time in the moulding shop is first considered, and a new method of calculation is described. The concepts of 'moulding dish size' and of 'extra volume' are introduced. The moulds are graded in terms of difficulty and examples given. By the use of 'casting box value tables,' calculations can be economically made and repetitive reckoning avoided.—R. J. W.

An Introduction to a Core Moulding Method by the Reorganization of a Large-Castings Foundry. F. Forti. (*Giesserei*, 1955, **42**, Sept. 1, 457–462). Works practice and the working programme are first described in general terms. The technique of core moulding methods is then described in detail and

illustrated examples of its use for difficult castings are given with advantages and disadvantages. Finally the organizational measures required to improve the working by the introduction of these methods is described.—R. J. W.

The Construction of an Impeller Moulding Device. W. Hagedorn. (*Giesserei*, 1955, **42**, July 21, 401–403). The construction of a contrivance for making impeller moulds is described in stages. The stages, which are well illustrated, include (*a*) the pattern, (*b*) the blade core box, (*c*) the scraper template, (*d*) the borehole core box, and (*e*) the core control gauge and construction base-plate.—R. J. W.

Castings from Chilled Iron in Boxless Moulds. B. A. Kremenetskii. (*Liteĭnoe Proizvodstvo*, 1955, (4), 8). [In Russian].

Pressure Pouring and Graphite Permanent Moulds Used in Production of Steel Car Wheels. H. H. Hursen. (*Amer. Found. Soc., Preprint*, No. 130, 1955). The development and operation of a pressure-pouring process using permanent graphite moulds is described and illustrated. In this process molten metal is forced up a refractory tube and into the graphite mould by compressed air. When the mould is full a stopper is inserted into the bottom of the mould which is then removed. The process is fully automatic and 20 moulds are cast from one filling of a 7-ton insulated ladle.—B. C. W.

Large, Intricate Shapes Made by Investment Casting. I. R. Kramer and D. V. Ludwig. (*Mat. Methods*, 1955, **41**, Apr., 106–109). The use of frozen Hg patterns to free investment casting from many of the size and thickness limitations imposed by wax or plastic patterns is described. Castings weighing 80–100 lb with dimensions in excess of 36 in. have been made by the new process.—P. M. C.

The Shaw Process of Precision Casting. (*Machinery*, 1955, **87**, Sept. 2, 577–584). Continuing a previous article (*Machinery*, vol. 81, pp. 768, 2nd Oct., 1952), a review of some typical examples of the current applications of the Shaw process of precision casting is given. The article illustrates the wide range of materials and shapes that can be cast by this process.—M. A. K.

The Manufacture of Pressure-proof Coatings for Shafts. F. Reile. (*Giessereitechn.*, 1955, **1**, Oct., 188–191). Castings 3 in. long, with diameters up to 500 mm and thicknesses up to 45 mm have been produced in the West by centrifugal casting. Results of experiments with other methods are given.—L. J. L.

Feeding Range in Shell Moulds. R. E. Morey, H. F. Bishop, and W. S. Pellini. (*Amer. Found. Soc., Preprint*, No. 136, 1955). The riser feeding range in ¼–1½ in. thick shell-moulded plate castings was investigated for eight different alloys (four ferrous, four non-ferrous) by radiography and hydraulic pressure tests. The feeding range for steel was 4·5 times the plate thickness and for nodular cast irons 6–6·5 times the plate thickness. Grey iron was essentially 'self-feeding.' It is concluded that feeding range data for conventional sand moulds may be applied to shell moulds.—B. C. W.

Development of Shell Moulding Technique. J. Fellows. (*Foundry Met. Treating*, 1954, **1**, Oct., 7–9, 11, 12, 25). The development of the shell moulding method of producing castings is reviewed and the process as it exists at present is described. An attempt is made to forecast some of the possible future developments of this unique technique.

The Production of Steel Castings by the 'Vimold' Process. C. R. Taralli. (*Bol. Assoc. Brasil Met.*, 1954, **10**, Oct., 381–384). [In Portuguese]. A description is given of the Vimold shell-moulding process as used at the Villares Steel Co., Ltd., São Paulo, Brazil. Criteria for pattern-making are discussed and each step in the shell-moulding process is described briefly.—P. S.

New B.S.A. Shell Moulding Foundry. (*Machinery*, 1955, **87**, Aug. 5, 297–304). The article describes a new hydraulically-operated shell-mould-making machine designed by the B.S.A. Co., Ltd., and built by Spares and Equipment Ltd. Pattern plates measuring up to 21 in. × 27 in. can be accommodated.

Engineering Methods in Practical Sand Problems:—Strength–Density Relationships. V. E. Zang and G. J. Grott. (*Amer. Found. Soc., Preprint*, No. 102, 1955). It is shown that, for a large number of different sand-mixtures, there is a linear relationship between the logarithm of the tensile, shear, or compressive strength and the density. A method is then described for calculating the shear and bond strengths, and the coefficient of intergranular friction from the tensile and compressive strengths. The calculated values are shown to correlate reasonably well with the measured values. The bond strength and coefficient of intergranular friction for several sands are plotted against sand density to determine the usable range of density.—B. C. W.

Principles of Agglomerating Processes. C. Ludwig. (*Amer. Found. Soc., Preprint*, No. 105, 1955). The principles involved in the packing of particles are outlined and the influence of size distribution, packing pressure, and surface tension is considered. The importance of correct mixing for obtaining the most suitable condition is stressed, and the action of binders is briefly discussed.—B. C. W.

A Critical Analysis of the Cylindrical Standard Test Specimen for Foundry Sands. W. Goetz. (*Amer. Found. Soc., Preprint*, No. 107, 1955). The strength–moisture and permeability–moisture curves obtained for clay-bonded moulding sands using the standard *AFS/DIN* cylindrical test specimen are considered in relation to similar curves which have been obtained with other test specimens. It is concluded that the apparently contradictory results arise from the failure to separate the independent effects of moisture and density, and a ternary diagram in which permeability and strength are plotted as functions of the volume taken up by sand, water, and air, is developed for this purpose.—B. C. W.

The Expansion of Moulding Sand with Increase of Temperature. R. G. Godding. (*Brit. C.I. Res. Assoc. J. Res. Dev.*, 1955, **6**, Aug., 23–29). The effect of moisture content, degree of ramming, and various additions on the expansion of natural and synthetic moulding sand with increase in temperature, has been studied.—B. G. B.

Effect of Mould Material on the Solidification Rate of Cast Metals. (*Inst. Brit. Found.*, 1955, June 20–24, Paper No. 1130). The work carried out by sub-committees T.S.21, 33, and 46 on the effect of mould materials on the solidification rate of cast iron and aluminium bronze is described and discussed. It was found that slush casting was not a satisfactory method for determining solidification rates, which was best done by pyrometric methods. With standardized conditions it was found that, at a given ramming density, the coarser the sand, the harder a mould was rammed, and the greater the moisture content of the sand, the greater the cooling power of the mould. In castings up to 1½ in. in thickness, coal dust caused some increase in cooling power.—B. C. W.

Spalling of Green-sand Moulds and its Relation to Casting Defects. H. Pettersson. (*Inst. Brit. Found.*, 1955, June 20–24, Paper No. 1129). An experimental investigation has been made into the spalling of green-sand moulds by a number of methods including direct observation of the behaviour of the upper mould wall. The mechanism of the spalling and the influence of sand variables on spalling are described. A detailed discussion is given of the factors affecting the spalling of moulds.—B. C. W.

Behaviour of Moulding Sands at High Temperatures. W. B. Parkes and R. G. Godding. (*Inst. Brit. Found.*, 1955, June 20–24, Paper No. 1128). An apparatus is described which enables a stress/strain curve to be determined on a sand test-piece which has been rapidly heated to the testing temperature. Results obtained on a synthetic silica–bentonite sand and on a natural red sand are presented and discussed and the effects of added wood-flour and coal dust are considered. It is suggested that spalling of the mould surface and hence scabbing on the casting will occur if the expansion of the sand on heating is greater than that at which the test-piece collapses. Results with olivine sand show that, if the expansion of the sand is reduced, the tendency to scabbing is also reduced. Possible methods of decreasing the expansion or of increasing the deformation of the heated sand are discussed and the practical applications of the results are considered.

Humidity as a Factor in the Production and Use of Foundry Patterns. M. Málek. (*Slévárenství*, 1955, **3**, (11), 359–362). [In Czech].

Coleman-Wallwork Automatic Mould Making Machine. (*Machinery*, 1955, **87**, Aug. 12, 358–362). The article describes a machine recently introduced by Coleman-Wallwork Co., Ltd., London. It is a fully-automatic mould-making machine known as the 'Moldmatic' and is capable of producing 240 sand-moulded halves per hour in boxes 34 in. long by 20 in. wide and 10 in. deep. The 'moldmatic' is intended for use with mechanized foundry installations.—M. A. K.

Use of Quick-Change Coordinated Plates in Machine Moulding. A. A. Shmelev. (*Liteĭnoe Proizvodstvo*, 1955, (5), 27–29). [In Russian]. The design and uses of various types of shaped insertion plates, capable of rapid fitting and replacement, are considered.

New Core-Binders Based on Synthetic Resins. J. Ornst.

(*Práce Československého Výzkumu Slévárenského* (*Appendix to Slévárenství*, 1955, **3**, (12)). [In Czech]. Experiments with new phenolic binders are described.—P. F.

New Knowledge on the Carbon Dioxide Method. W. Schumacher. (*Giesserei*, 1955, **42**, Nov. 24, 653–659). Investigations on the thermal conductivity and strength properties of sand mixtures hardened with carbon dioxide are described and their influence on the decay of moulds and cores is discussed. The marked influence of the rate of cooling is noted. Several new applications are illustrated which show the versatility and economy of the method.—R. J. W.

Low Pressure Casting of Steel in Graphite Moulds. H. H. Hursen. (*Machinery*, 1955, **87**, Nov. 18, 1187–1192). Methods of casting steel wheels for railway wagons by The Griffin Wheel Co., Chicago, are described. Molten metal is forced upwards into a graphite mould by means of compressed air applied to the surface of the steel in a ladle beneath the mould. Due to the high rate of heat transfer, solidification of a 700-lb casting can be achieved in 5 min. Wheel hub centres are cut out by oxy-acetylene torch and normalizing and machining complete the operations.—M. A. K.

Further Development of Shoulder Rod Iron Chills. A. Diez. (*Giesserei*, 1955, **42**, Oct. 27, 610–611). The construction and use of shoulder iron moulds for rod specimens of an improved design are described. Comparative experiments on rods of various alloys so produced indicated a considerably reduced spread on the values of the strength properties.—R. J. W.

A Template for a Spiral Housing of a Water Turbine Moulded in Sand. F. Paschke. (*Giesserei*, 1955, **42**, Sept. 15, 537–539). The preparation of moulds for a 9000-kg spiral housing for a water turbine is described. The bottom casting box has overall dimensions of 500 cm × 580 cm. The manufacture of the core for the casing is described and illustrated, as is the preparation of the upper casting. Finally the blackening and drying of the moulds are described.—R. J. W.

A Case Study in a Heavy Foundry. J. Souter. (*Brit. C.I. Res. Assoc. J. Res. Dev.*, 1955, **6**, Aug., 2–10). The results are given of an operational research study to determine improvements which could be effected in the productivity of a heavy core-shop attached to a jobbing foundry engaged in the manufacture of heavy electrical castings.—B. G. B.

Centrifugal Casting. (*Metal Ind.*, 1955, **87**, Oct. 21, 347–349). A brief description with illustrations is given of the equipment and production methods employed at the Sutton-in-Ashfield works of Sheepbridge Alloy Castings Ltd. Tubes, sleeves, and liners are spun-cast in stainless steel and other ferro-alloys.—P. M. C.

Investments for Precision Casting: Alternative Materials. D. E. Brooks. (*Iron Steel*, 1955, **28**, Oct., 481–482). A summary is given of American work carried out to find suitable alternative materials to replace the normal investment materials in short supply. The data show that low-price foundry sands can be used as the refractory, and the results obtained with alumina-base cement, chemical, and plaster binders are compared.—G. F.

Six Factors to Consider in Selecting Your Investment Casting Alloy. F. K. Iverson and J. B. Meierdirks. (*Precision Met. Mold.*, 1955, **13**, Oct., 52–53, 85–86). The following factors in the selection of raw material for investment casting are briefly discussed: (1) Analysis, (2) Form, (3) External cleanliness, (4) Metal purity, (5) Availability, and (6) Metal cost.

Progress in the Manufacture and Use of Centrifugal Steel Castings. F. Pölzguter. (*Giesserei*, 1955, **42**, Sept. 15, 493–500). The principles behind the different methods of centrifuging are first described and are followed by an account of the historical development of the method and the present patent position. The production of steel tubes by horizontal and vertical centrifugal casting is then described. This section is well illustrated with photographs, diagrams, and micrographs. Centrifugal mould casting is briefly described. The range of uses for centrifugal casting is given.—R. J. W.

Shell-Moulding of Cylinders with Cooling Ribs. R. Kloda. (*Slévárenství*, 1955, **3**, (11), 352–355). [In Czech].

The A.P.V.-Paramount Shell Moulding Foundry. (*Machinery*, 1955, **87**, Nov. 25, 1224–1244). Some details of the layout and operation of a shell-moulding plant installed two years ago at the works of A.P.V.-Paramount Ltd. are given. Examples of machining economies obtained with this method over previous box-moulding technique are discussed.—M. A. K.

Shell Moulding in Canada. E. W. Chapman. (*Canad. Metals*, 1955, **18**, Nov., 39–44). An account of recent developments in the use of shell moulding in Canada is presented. Equipment in operation is described.—B. G. B.

Shell Moulding Gains Over Sand. (*Canad. Metals*, 1955, **18**, Aug., 33–36). An account is given of the successful adoption by Fairbairn–Lawson, Combe Barbour Ltd., of Leeds of the shell-moulding process.—B. G. B.

Gases in Cast Iron. E. B. Simonsen. (*Tidsskr. Kjemi, Bergvesen Met.*, 1955, **15**, (1), 6–13). [In Norwegian]. Based on a review of 22 recent papers dealing with gases in under-eutectic cast iron, the article deals with factors causing porosity, abnormal contraction, and poor mechanical strength. An examination is made of opinions expressed regarding the effects of hydrogen, oxygen, and nitrogen, their volume ratios, gas sampling technique, and the origins of such gases.—G. G. K.

Oxygen Content of Grey Cast Iron Increases With Lime. R. C. Williams and H. W. Lownie. (*Amer. Found. Soc., Preprint*, No. 22, 1955). It is pointed out that many of the oxygen contents of up to 0·5% which are reported in the literature for cast iron have been obtained on specimens exposed to the atmosphere for long periods, and that thermochemical data indicate that the oxygen content of grey cast iron with normal silicon contents should only be between 0·001 and 0·004%. Vacuum fusion oxygen analyses were made at intervals of up to a year after casting on three flake and one nodular iron, and it was found that the oxygen content of all the irons increased in this period by a factor of between 2 and 4. The increase was not due to surface rustings, as the specimens were grit-blasted before analysis, but must in some way be connected with the graphite, as the rate of oxygen absorption increased with the size of the graphite flakes connected to the surface, being least for the nodular iron.—B. C. W.

Dissolved Gas in Liquid Cast Iron. W. Y. Buchanan. (*Amer. Found. Soc., Preprint*, No. 25, 1955). A description is given of a simple method for collecting the gas evolved from liquid cast iron, using a cast-iron bell connected to an aspirator and dipping below the metal surface. The results obtained for gas composition, total gas volume, and rate of gas evolution are presented and discussed. The effects of rust, mill scale, and soda ash on gas evolution and composition were determined and it was found that mill scale reduced, and soda ash increased, the total volume of gas evolved.

New Method for Determining the Fluidity of Alloys by Vacuum Suction. Yu. A. Nekhendzi, N. G. Girshovich, and E. I. Egorov. (*Zavodskaya Laboratoriya*, 1955, **21**, (1), 66–68). [In Russian]. The drawbacks of existing methods for determining the fluidity of liquid alloys are discussed and a technique in which the liquid is sucked up a quartz tube connected to an evacuated vessel is described. The tube is immersed to a depth of 50–70 mm, and suction is applied after 10–15 s. Fluidity is estimated from the length of tube filled with metal less the depth of immersion, the latter being indicated by a copper wire fixed to the outside of the tube. Results obtained with iron (3·02% C, 1·96% Si) at 1400° C are presented.

Casting Defects due to Sand. Mould Resistance to Thermal Stresses. H. Pettersson. (*Jernkontorets Ann.*, 1955, **139**, (9), 709–762). [In Swedish]. Details are given of studies of the resistance of green and dry sand moulds towards the heat given off by the molten metal, these being undertaken by means of ordinary casting experiments with closed moulds, but mainly by using moulds in which a 10-mm gap between the two halves facilitated observation of the mould wall. The paper deals with the influence of heat intensity, sand composition, and mould hardness on the spalling resistance of the mould, methods of determining the thermal expansion of different moulding sands, and measurements of temperature distribution in the moulds.—G. G. K.

Gases in Cast Iron. H. Morrogh. (*Inst. Brit. Found.*, 1955, June 20–24, Paper No. 1137). The effects commonly attributed to oxygen, hydrogen, and nitrogen in cast iron are outlined, and the methods developed for the sampling and analysis of cast iron and pig iron for these elements are described. Gas analyses from a wide range of commercial irons are summarized and their significance is discussed. Recent research at the B.C.I.R.A. on the effects of oxygen and nitrogen on cast iron is reviewed and work on the accurate estimation of hydrogen is considered.—B. C. W.

Widmanstätten and Other Abnormal Graphite Forms in Cast Iron. E. B. Simonsen and F. Brown. (*Inst. Brit. Found.*, 1955, June 20–24, Paper No. 1131). A number of abnormal forms of graphite which may occur in grey cast iron are

described and the conditions of their development briefly discussed. Experimental melts indicate that small additions of lead may give Widmanstätten graphite. It is suggested that impurities may affect the graphite–metal surface energies and thus give rise to different forms of graphite.—B. C. W.

REHEATING FURNACES AND SOAKING PITS

Modern Designs of Soaking-Pits. L. G. Aksel'rud. (*Stal'*, 1955, (3), 268–273). [In Russian]. Many aspects of soaking-pit design are considered, comparisons being made with American practice. Possibilities of using the heat of the exit gases and of automatic regulation and the heating power of pits are compared for the various types, and pit-designs and performances in new blooming-mills in the U.S.S.R., the rate of heating, productivity, fuel- and refractory-consumptions, and methods of slag-removal are reviewed.—S. K.

Soaking Pits. Types, Construction, Use and Maintenance, Recuperation, and Control. Courdille. (*Centre Doc. Sidér., Circ. Inform. Tech.*, 1955, **12**, (11), 2161–2174). Bottom-heated pits in which combustion gases leave at the sides, circular pits with tangential heating, single-burner pits with the burner in the upper part, and similar pits with two burners are all described and performances are briefly compared. 'Jet-pump' and Escher recuperators are mentioned and control is discussed. Reheating of special steels as practised at Atlas Steels is outlined.—T. E. D.

Insulation of the Bottom Pipes of Continuous Furnaces. V. P. Merkhilevich and I. S. Omel'chenko. (*Stal'*, 1955, (2), 183–184). [In Russian]. Methods of insulating water-cooled pipes in continuous-furnace bottoms are considered, with brief mention of the effect on furnace operation.—S. K.

Construction and Operation of Modern Forging Furnaces. K. Niemeyer. (*Stahl u. Eisen*, 1955, **75**, Aug. 11, 1029–1035). The construction of forging furnaces still depends on individual experiences and this results in furnaces of the same size having different numbers and arrangements of burners. It is important that there should be good circulation and turbulence of the hot gases, and a marked reduction in the number of burners should be possible. The planning and layout of the recuperators require special attention in view of the discontinuous operation of the furnace. The determination of pre-warming and holding times is difficult and standard charts for fuel flow and surface temperatures should therefore be established.—J. P.

Induction Heating Steel with 60 Cycles. C. D. Kramer. (*Trans. Amer. Inst. Elect. Eng.*, 1954, **73**, Part II, 353–357). Theoretical considerations governing the low-frequency induction heating of steel billets are discussed. It is shown that round or square sections greater than 10 in. across or slabs greater than 7 in. can be heated successfully at 60 cycles; higher frequencies are required for smaller sizes.

Gas Furnaces Used for Aluminium Coating Process. A. Q. Smith. (*Indust. Heating*, 1955, **22**, July, 1380–1390). Furnaces used in the 'Alumibond' process in which aluminium alloys are cast on to steel liners, and the 'Alumicoat' process, a hot dip process, are described.—A. D. H.

Induction Heating for Hot Forming. F. T. Chesnut. (*Indust. Heating*, 1955, **22**, Mar., 492–504; 654–668). The author gives a general account of heating by low-, high-, and dual-frequency methods. Design of electrical and control equipment and coil design are discussed. The application of low- and high-frequency methods to heating for forging is considered and several industrial installations are described.—A. D. H.

HEAT-TREATMENT AND HEAT-TREATMENT FURNACES

Heat Treat Controls Boost Job Shop Quality. W. G. Patton. (*Iron Age*, 1955, **176**, Oct. 20, 103–106). The system of control in a commercial heat-treating shop handling 20–30 tons of work per day is described. The equipment is standard but versatile, and close control is possible.—D. L. C. P.

Additional Heat Treatment of Structural Steel. Z. M. Kalinina, R. I. Kolyasnikova, and E. M. Poznyakova. (*Stal'*, 1955, (2), 164–167). [In Russian]. The results are analysed of experiments on the use of supplementary heat-treatment for improving the mechanical properties of alloy constructional steels under mass-production conditions in metallurgical works.—S. K.

The Heat Treatment of Carburising Steels. F. P. Heard. (*Australian Inst. Metals: Australasian Eng.*, 1955, Apr. 7, 44–50). The author deals with the selection of carburizing steels and discusses recent developments in their practical heat-treatment. Emphasis is laid on the trend to lower-alloy higher-carbon steels and on the increased use of controlled atmospheres, which has been made possible by better knowledge of metal to atmosphere equilibria and by better instrumentation. (8 references).—P. M. C.

Heat Treatment Practice in Relation to the Dimensional Stability of Steel. N. J. Sheppard. (*Australian Inst. Metals: Australasian Eng.*, 1955, Mar. 7, 46–56). The problems of distortion associated with heat-treatment practice, and of long-term dimensional stability are reviewed. The notable influence of component design and type of material on dimensional stability is discussed, and an outline of the role of residual stress conditions and relief of same is given. The existence of tetragonal martensite in untempered structure which may lead to cracking makes tempering essential. Factors affecting dimensional stability in case-hardening, gaseous-nitriding, and induction-hardening processes are outlined. Troubles associated with retained austenite, which may subsequently transform to martensite, are discussed and cycles for the removal of retained austenite are indicated. (48 references).—P. M. C.

Practical Applications of Furnace Atmospheres. C. A. Mueller. (*Metal Treating*, 1955, **6**, Mar.-Apr., 2–7, 28). Methods and equipment for the production of endothermic and exothermic atmospheres and for the dissociation of ammonia are described. The applications of these various atmospheres in various heat-treating processes are discussed.

Commercial Heat Treating Shops In France. B. Jousset. (*Metal Treating*, 1955, **6**, May-June, 12, 14–16, 37). The most important commercial heat-treating shops in France (27 in number) are listed. The operation of 'integrated' shops, attached to or connected with large works, is discussed, and general trends in heat-treatment practice in France are briefly reviewed.—P. M. C.

An Unusual Commercial Heat Treating Plant. (*Metal Treating*, 1955, **6**, July-Aug., 12–13, 26). Furnace equipment at the Bosworth Steel Treating Co., Detroit, is described.

Basic Principles of Electrode Type Salt Bath Furnaces. L. B. Rosseau. (*Metal Treating*, 1954, **5**, May-June, 2–4, 15). The basic advantages of salt-bath heating for heat treating are outlined, and constructional features of immersed-electrode type baths are discussed, with data relating to their use. The desirability of an incorporated salt-purification system is emphasized.—P. M. C.

Individual Heat Treatment of Quality Rolls. (*Indust. Heating*, 1955, **22**, May, 976–978). The heat-treatment of cast rolls by normalizing, annealing, and quenching and tempering is briefly described.—A. D. H.

Versatile Heat Treating Furnace Installed by Northwest Fabricator. H. F. Knab. (*Indust. Heating*, 1955, **22**, Apr., 750–756, 835). A gas-fired bogie hearth furnace 20 × 20 × 30 ft, capable of operation in the temperature range 600–2100° F, is described.—A. D. H.

Definition of the Action of Protective Gases. E. Schwarz-Bergkampf. (*Radex Rundschau*, 1955, July, 499–505). [In German]. Principles are defined and discussed for the use of controlled protective atmospheres in the production and heat-treatment of metals, particularly of iron. Uses of active as well as of inert gases are described, and the calculation of gas composition requirements is explained.—E. C.

Heat Treatment Problems. Their Solution in Electric Furnaces. R. O'Donoghue. (*Elect. Rev.*, 1955, **157**, Aug. 26, 383–386). Examples are given of the application of electric furnaces to the heat-treatment of metals.—L. D. H.

A Review of Salt Bath Carburizing. E. N. Case. (*Metal Treating*, 1955, **6**, May-June, 18–19; July-Aug., 8–10, 33). The use of cyanide salt baths for case carburizing is described, and the relationships between case depth, time, and temperature are briefly reviewed. The use of catalysts such as calcium, barium, and strontium compounds is discussed.

Case Hardening and Heat Treatment of Steel in "Cassel" Salt Baths. D. Thomas. (*Eng. Insp.*, 1955, **19**, Summer, 82–91). "Cassel" salts, which are products of I.C.I. Ltd., are available in many types, and the author presents data for bath composition, temperature, time, and depth of case together with microphotographs for both shallow and deep case carburizing. The treatment of high speed and semi-high speed steels in salt baths is briefly described.—P. M. C.

A Method of Carburizing and Direct Martempering Suitable for Small Parts. G. Veronesi. (*Ing. Mecc.*, 1955, **4**, Feb., 33–38). [In Italian]. The author discusses the difference between marquenching and martempering and explains the trend of Bain's curve in case hardened steels for the differing carbon contents encountered from the top hardened skin down to the softer core. The different methods of quenching after carburizing are listed and the drawbacks which most methods present are examined. The author claims that the method of direct martempering from carburizing temperature produces a bainitic martensitic skin structure and a martensitic core which offers the advantages of a simple and satisfactory process while reducing the possibility of distortion. Theoretical considerations which are based ou Bain's curves are fully discussed.—M. D. J. B.

Gas Carburizing—Case Hardening in Carburizing Atmosphere. G. Limentani. (*Ing. Mecc.*, 1955, **4**, Aug., 41–45). [In Italian]. The author reviews the advantages of gas over pack carburizing and describes the mechanics of atmosphere carburizing in which the carburizing atmosphere is composed of a carrier and an enriching gas. It is suggested that natural gas is preferable to carbon monoxide. The author shows the merits of using the dew point as a means of controlling the carburizing process and goes on to describe some modern fully instrumented carburizing furnaces in the U.S.A.—M. D. J. B.

Induction Hardening of Long Workpieces. G. W. Seulen. (*Z.Vd.I.*, 1955, **97**, Sept. 1, 869–876). Progressive hardening by means of a short induction coil traversing the length of the workpiece is recommended on account of greater economy and smaller distortion. Constructional details of coils and associated quenching appliances are described. The article also contains data of the relation between traversing speed, induction frequency, and depth of hardening. Machines for progressive hardening of cylindrical shapes, including rolls and shafts, are described.—J. G. W.

A Review of Some Factors Influencing Nitriding Practice. G. L. Cox. (*J. B'ham. Met. Soc.*, 1955, **35**, June, 213–230).

Correct Bath Nitriding of High Speed Steel Tools. A. Rühenbeck. (*Metalloberfläche*, 1955, **9**, May, A65–A68). The process of bath nitriding, previous experiences with the application of the method, and the chemical and metallurgical processes taking place are described, and the advantages and disadvantages are discussed. It is concluded that bath nitriding can only be used to the best advantage if the high speed tool is first hardened and tempered in neutral salt baths before nitriding.—L. D. H.

Application of Nitriding To Hot Forging Dies. A. F. Sherys. (*Metal Treating*, 1955, **6**, Jan.-Feb., 14, 16–17). The application of surface hardening by the nitriding process to forging dies is advocated as a means of improving die life. The characteristics of nitrided surfaces are briefly reviewed and it is pointed out that by suitable stopping-off techniques the actual forging impression alone can be treated to a file-hard condition. Increased forging die life as high as 400% is quoted.—P. M. C.

A New Universal Progressive Hardening Machine. (*Wild Barfield Heat Treatment J.*, 1955, **5**, Sept., 10). A brief description is given of a machine designed for use with Wild Barfield A.H.F. high-frequency equipment to harden steel bar progressively.—D. H.

Bright Hardening and Bright Tempering of Corrosion-Resistant and High-Temperature Alloys. T. W. Harker. (*Metal Treating*, 1955, **6**, May-June, 2–4). Details are given of specialized heat-treatment procedures for bolts, nuts, and other fastenings for high duty service. These include annealing and precipitation hardening of austenitic alloys, and hardening and tempering of martensitic alloys. In all cases protective furnace atmospheres are used.—P. M. C.

Commercial Bright Hardening of Stainless Steels. F. Hunter. (*Metal Treating*, 1955, **6**, July-Aug., 20–22). A gas-fired muffle-type furnace is described which is successfully used for air-hardening and annealing stainless steels without any detriment to surface brightness in an atmosphere of dissociated ammonia. The furnace operates on a continuous cycle.—P. M. C.

FORGING, STAMPING, DRAWING, AND PRESSING

A 2,000 T Forging Press. (*Overseas Eng.*, 1955, **29**, Oct., 96). A 2000-ton four-column forging press recently installed by Babcock and Wilcox Ltd., is described which is used for making boiler drums. The press will take the heaviest rolled plate of max. width of 12 ft 6 in. and is suitable for pressing drum ends from 12 ft 6 in. circular blanks 7 in. thick. Approximate overall height of press above floor level is 18 ft 9 in.

Two Decades of Progress in The Forging Industry. H. J. Merchant. (*Australian Inst. Metals: Australasian Eng.*, 1955, July 7, 56–67). A general description is given of the outstanding developments made in forging practice during the past twenty years. The author deals with materials and forging metallurgy, pressing, impacting, and upsetting equipment, and die production and tooling. (24 references).—P. M. C.

Open Shape Forging. E. Pflaume. (*Metallurgie*, 1955, **5**, Aug., 245–250). Materials, their defects, ways and means of eliminating defects, furnace construction, warming up, presses, hammers, forging processes, methods of non-destructive testing, and the economics of forging are discussed.

Metal Gathering: What It Is and How It Works. W. E. Achor. (*Iron Age*, 1955, **176**, July 14, 99–102). It is explained that the metal gathering process accumulates plastic metal under pressure at the ends or in the middle of tubing or bar stock. A portion of the stock is electrically heated, and the required length consolidated by hydraulic ram. The gathered part can then be forged and machined to shape. Most carbon and alloy steels are amenable to this treatment.

Simplify Sectional Dies to Save Material and Labour. F. Strasser. (*Iron Age*, 1955, **176**, Oct. 13, 102–104). Information on the advantages and construction of sectional dies for blanking, punching, cutting off, bending, forming, and drawing is given.—D. L. C. P.

Can Controlled Temperature Rolling Improve Drawability? N. P. Goss. (*Iron Age*, 1955, **176**, Aug. 11, 100–102). Experiments indicating that controlled temperature rolling improves deep-drawing properties of stainless steel sheet are described. In the process sheet is rolled at 500° F, producing a randomly orientated grain structure, as a result of which transverse and longitudinal properties are more compatible.—D. L. C. P.

A New Deep-Drawing Process. V. Kovačec. (*Maschinenwelt Elektrotechnik*, 1955, **10**, Sept., 257–258). A new hydraulic machine is described suitable for deep-drawing alloy steel sheet, including manganese steels.—L. D. H.

Negative Lubricating Action of Some Liquid Media in the Deep Drawing of Metals. S. Ya. Veiler. (*Doklady Akad. Nauk SSSR*, 1952, **83**, (5), 709–712). [In Russian]. The influence of some lubricants on tangential and normal drawing forces during drawing specimens of carbon steel (0·09–0·15% C) as well as the effect of negative and positive lubricants on deformation and surface hardening of the metal were investigated. On the basis of results obtained some conclusions are drawn as to the nature of the action of lubricants on tangential and compressive (normal) drawing forces.—V. G.

Automatic Scrap System Handles 55 Tons Per Hour. W. G. Patton. (*Iron Age*, 1955, **176**, July 28, 68–70). An automatic scrap disposal system for removing sheet scrap from the presses and turning out 1000-lb bales, is described. The triple compression baler cycles every 30 seconds.—D. L. C. P.

Cold Extrusion is Shaping Up. J. E. King. (*Steel*, 1955, **137**, Oct. 3, 70–71). The type of job suitable for cold extrusion, the materials available and the savings possible are outlined.

Tooling for Cold Steel Extrusion. J. F. Leland. (*Machine Shop Mag.*, 1955, **16**, Aug., 469–475). Savings of 40–60% are claimed for cold extrusion of steel over other methods of fabrication. The author outlines the essentials of tooling and procedure needed for successful exploitation of the process.

ROLLING-MILL PRACTICE

Steel Mill Modernization. (*Elect. Rev.*, 1955, **157**, Oct. 7, 685). The electrical equipment for the rolling mills now being reconstructed at the Workington branch of the United Steel Companies is described.—L. D. H.

A Suggestion for the Standardization of Tables used in the Roll-Turning Shop. O. Müller and G. Beier. (*Metallurgie*, 1955, **5**, June, 203–205). So-called "percentage tables" are used in roll turning shops when turning metallurgical rolls from drawings. These tables differ from plant to plant: the authors advocate the introduction of a standard edition of the tables.—L. J. L.

Hard Metal Rolls. J. Hinnüber and H. D. Dietze. (*Tech. Mitt. Krupp*, 1955, **13**, Sept., 118–120). The design and construction of hard metal rolls are outlined, and the advantages in their use are explained. The main physical properties of hard metals suitable for this purpose are tabulated. Some

examples of the use of the rolls are given, and the incidence of cracks in the rolls due to overloading is discussed, with reference to a calculation of the stresses involved.—L. D. H.

Some Experiences with Plastic Bearings in Rolling Mill Rolls. F. Nuhlíček. (*Hutník*, (Prague), 1955, **5**, (10), 299–302). [In Czech].

Rolling Mill Bearing Seals: Design and Performance of the Walkersele. (*Brit. Steelmaker*, 1955, **21**, June, 188–190). The design considerations and other requirements of a good bearing seal arrangement are described, and details are given of the " Walkersele," developed by James Walker & Co. Ltd., to meet these requirements. An indication is given of its performance in practice.—G. F.

Hot Rolling of Steel Strip. S. Ryhagen. (*Jernkontorets Ann.*, 1955, **139**, (9), 617–708). [In Swedish]. After comparing the production and consumption of steel plate in Sweden and nine other countries, the author discusses developments in strip rolling mills with special reference to the hot Steckel mill at Fagersta, the Sandviken and Hagfors semi-continuous mills, and the Morgan mill at Domnarfvet. Measures necessary to adapt a modern rolling mill to the rolling of small orders are discussed. Other aspects dealt with include the possibility of using statistical methods for controlling strip dimensions, and factors causing defective strip surfaces.—G. G. K.

Friction in Cold Rolling and Its Governing Variables. P. W. Whitton. (*Australasian Eng.*, 1955, Aug. 8, 81–87, 121, 123, 125, 127). An examination is made of the effect of load, speed, surface finish, and work hardening upon the value of the mean coefficient of friction in cold rolling. Copper, mild steel, brass, and aluminium were tested with various lubricants, the coefficients of friction being determined by the method previously published by the author. The actual friction mechanism is examined via surface finish tests. (10 references).

The Serviceability of Various Emulsions for the Cold Rolling of Steel Strip. G. Dallmeyer. (*Stahl u. Eisen*, 1955, **75**, Aug. 25, 1119–1120). The author discusses the criteria to be observed in an attempt to develop better, or improve existing, rolling oils, in particular, the interdependence of various properties such as stability at high pressure, long life, rust protective power, self-cleaning properties, freedom from depositing dirt on strip or machines, etc.—J. P.

Cold Rolling of Phosphated Steel Strip. W. Lueg and K-H. Treptow. (*Stahl u. Eisen*, 1955, **75**, Aug. 25, 1085–1092). Steel strip (0·1 and 1·1% C) with and without phosphate treatment has been cold rolled with rape-seed and mineral oils as lubricants on a twelve-roll train with 10-mm dia. rolls and on an experimental mill with 199-mm rolls. Under some conditions the presence of a phosphate layer reduced rolling force and power, though the effect was less with the higher carbon material, the difference being attributable to the varying behaviour of the phosphate layers at low- and high roll pressures. The tensile properties of the rolled strip showed that the phosphated material was more work-hardened than the non-treated, once more due to the different conditions in the roll gap.—J. P.

Main Drives for Reversing and Continuous Hot-Rolling Mills. G. Zinkernagel. (*A.E.G. Progress*, 1955, (2), 48–54). The electrical equipment for blooming mills and continuous rolling mills must meet unusual and exacting requirements. Various types of drive in use are described.—B. G. B.

Electric Drives for Wide-Strip Reversing Rolling Mills. H. E. Knobloch. (*A.E.G. Progress*, 1955, (2), 54–59). The design of the electric drives for Steckel-type rolling mills is discussed. Particular reference is made to a mill constructed by A.E.G. 15 years ago, and to a finishing mill train of a wide strip rolling mill modernized in 1953.—B. G. B.

Three Phase Auxiliaries and Their Control Gear in Rolling Mills. O. Franz. (*A.E.G. Progress*, 1955, (2), 91–97). An investigation is made into the characteristics of three-phase drives for use in rolling mills. Examples from a 6½ ft medium and heavy plate mill of modern construction are used to indicate the extent to which three phase drives may be successfully used.—B. G. B.

Rectifier Installations for Rolling Mill Drives. F. Hölters. (*A.E.G. Progress*, 1955, (2), 110–127). A summary is first made of the operating conditions of rectifiers in rolling mills. The selection of electrical circuits, type of rectifier, and design of the control gear are discussed in detail.—B. G. B.

Rolling Mill Auxiliaries. J. Lapper. (*Metal Ind.*, 1955, **86**, Mar. 18, 207–209). Descriptions are given of several machines designed to facilitate the operation of single-stand rolling mills. These units, made by Auxiliary Rolling Machinery Ltd., West Bromwich, include a 3-roll coiler, a coil-opener which feeds the flattened end of a coil into the mill, a 7-roll flattening machine for sheets, and a strip slitting machine which does not damage or mark the strip.—P. M. C.

The Application of Hard-Facing Alloys in Rolling Mills. Chouteau. (*Centre Doc. Sidér., Circ. Inform. Tech.*, 1955, **12**, (11), 2175-2182). An outline is given of the use of hard-facing alloys in situations where resistance to abrasion, both hot and cold, is required. Examples are shown of the use of such alloys in guides for rod and bar mills of various types.

Organization of Repairs on Rolling-Mill Equipment during Stoppages. V. S. Koloskov and V. I. Volobuev. (*Stal'*, 1955, (2), 184–185). [In Russian]. The recording and organization of repairs on rolling mill equipment during planned and unplanned stoppages are described.—S. K.

Modern Development of Tube and Bar Straightening Machines. F-W. Neumann. (*Stahl u. Eisen*, 1955, **75**, Aug. 25, 1112–1113). The development of the high speed, high output straightening machine with two driven and five pressure rolls and its advantages and disadvantages are discussed.—J. P.

A New Roll-Setting Recorder. A. Unčovsky. (*Hutnické Listy*, 1955, **10**, (11), 676–679). [In Czech]. Telemechanical equipment utilizing two coupled Selsyn-type motors is described.—P. F.

An Automatic Roll-Adjustment Indicator. A. Winkler. (*Hutnické Listy*, 1955, **10**, (11), 673–675). [In Czech].

MACHINERY FOR IRON AND STEEL PLANT

Accident Prevention in Steelworks. A. Elson. (*Iron Steel*, 1955, **28**, Nov., 527–529). The author discusses how the job of preventing accidents in steelworks should be tackled. The principal causes of injury are mentioned and suggestions for guarding against them are given.—G. F.

No Chain Failures in this Foundry. J. Hogans. (*Mod. Castings Amer. Foundryman*, 1955, **28**, Aug., 62–65). The ordering, testing, inspection, and repair procedures which are used at the Beloit works of Fairbanks, Morse and Co., for all chain lifting devices, are illustrated and described.—B. C. W.

Specification for Heavy Duty Electric Overhead Travelling Cranes for use in Iron and Steel Works. (*Brit. Iron Steel Res. Assoc.*, 1955, Sept.). A complete specification for this type of crane which has been prepared by the Association is given.—B. G. B.

Automatic Weighing Machines for Granular Material. Z. Janoušek. (*Elektrotechnik*, 1955, **10**, (2), 59–62). [In Czech]. The construction and performance of a new Czechoslovak weighing machine for industrial use with granular materials are described.—P. F.

Dynamic Forces in Cranes. J. Götzlinger and S. Johnsson. (*Acta Polytechn., Mech. Eng. Series*, 1955, **3**, (7), 1–34). A comparison is first made between the American, German, and Swedish Standards for calculation of the dynamic forces in cranes. Formulæ for the maximum hoisting factor and travelling factor are deduced and checked experimentally.

Maintenance of Lifting Equipment. (*PACT*, 1955, **9**, Oct., 377–381). A system is outlined in which all lifting equipment on a works is listed, and description, characteristics, and inspection and maintenance schedules of each item are entered on separate cards.—T. E. D.

LUBRICATION

The Relation between Friction and Wear for Boundary-Lubricated Surfaces. E. Rabinowicz. (*Proc. Phys. Soc.*, **B**, 1955, **68**, Sept. 1, 603–608). A linear relationship between (wear rate)$^{2/3}$ and friction coefficient explains data from experiments in which metal transfer and loose wear were measured, including Neely's work (Inst. Mech. Engrs.: *Proc. Disc. Lubrication and Lubricants*, 1937, **2**, 378) on bronze in contact with steel.—K. E. J.

Apparatus for the Comparative Evaluation of the Colloidal Stability of Greases. K. I. Klimov, V. V. Sinitsyn, and E. V. Aleeva. (*Zavodskaya Laboratoriya*, 1955, **21**, (1), 106–108). [In Russian]. A simple and convenient method has been developed for evaluating the storage life of greases, based on the measurement of the amount of oil which pressure removes from the grease.—S. K.

Apparatus for Characterizing the Temperature Dependence of Lubricating-Oil Viscosity. A. N. Kislinskii and M. M.

Kusakov. (*Zavodskaya Laboratoriya*, 1955, **21**, (1), 102–105). [In Russian]. The apparatus for studying the viscosity of lubricants at various temperatures described is based on a modification of the falling-sphere method.—S. K.

Task of the Lubrication Engineer. B. R. Davies. (*Brit. Steelmaker*, 1955, **21**, Sept., 288–293). The author reviews a range of lubrication necessities in steelworks, referring particularly to rolling-mill installations. Improvements in roll-neck bearing design, including plain metal, fabric, roller, and fluid film bearings, are discussed and their lubrication systems are described.—G. F.

WELDING AND FLAME-CUTTING

Some Experiments on High-Manganese Arc-Welding Rods. D. R. Dhanbhoora, S. Visvanathan, and S. N. Anant Narayan. (*Tisco*, 1955, **2**, Oct., 173–177). The authors describe welding experiments from which it is concluded that (1) ferro-manganese in the flux coating of standard structural grades of electrodes may be replaced successfully by manganese in the core wire, provided metallic iron is incorporated in the flux coating, and (2) it may be necessary to make a metallic addition to the flux coating in order to stabilize the arc. This addition plays one of the roles that the ferro-alloy normally does when used in flux coatings.—L. E. W.

Initial Characteristics of Chromium–Nickel Steel Weld Metals. J. Henschkel. (*Welding J.*, 1955, **34**, Oct., 484s–504s). A study was made of the temperature sensitivity of iron-base chromium–nickel steel metal-arc weld metals under tensile stress. It is shown that some compositions and structures exhibit a low order of ductility in the as-deposited condition over a temperature range which is centered at 1800° F giving rise to cracking.—U. E.

Problems of Soldering and Brazing. J. Colbus. (*Schweissen u. Schneiden*, 1954, **6**, Special Issue, 140–147). The ' binding-temperature ' in soldering or brazing has been defined. The effect of gap width on the strength of the joint is shown to be mainly an effect of state of stress. The use of non-corrosive soldering fluxes is discussed.—U. E.

The Behaviour of Corrosion Resistant Chrome–Nickel Steel in Welding. F. Rapatz. (*Schweissen u. Schneiden*, 1954, **6**, Special Issue, 128–133). An investigation is described on stress corrosion cracking and on intercrystalline corrosion of welded 18/8 steels. It is shown that stress corrosion cracking can be avoided by normalizing at 900° C. Hot shortness is mainly due to the wrong choice of welding electrodes.—U. E.

The Present Position of Thermit Welding. W. Ahlert. (*Schweissen u. Schneiden*, 1954, **6**, Special Issue, 116–124). The metallurgy and properties of ' Thermit-welded ' iron is discussed. Practical applications are illustrated.—U. E.

Testing Welded Metal Joints. S. W. Hollingum. (*Metal Ind.*, 1955, **87**, Nov. 11, 401–404; Nov. 18, 429–432). The author describes welder's competency tests, and then discusses various methods of testing welded joints for cavities, porosity, cracks, etc. Radiographic tests using X-rays and radioactive isotopes, ultrasonic examination, and magnetic crack detection are all dealt with in some detail.—P. M. C.

Welding Fumes: Their Effects and Methods of Control. A. Bell. (*New Zealand Eng.*, 1955, **10**, Aug. 15, 263–268). Some occupational illnesses caused by welding fumes are discussed, and different systems of local exhaust ventilation are described and illustrated. Respirations are effective protection, but are all too frequently discarded. (9 references).—P. M. C.

Some Fatigue and Tensile Tests on Welds. P. F. Tomalin. (*New Zealand Eng.*, 1954, **9**, Nov. 15, 381–385). Experiments are described in which an attempt was made to determine the influence of defects on the fatigue strength of electric-arc butt welds in $\frac{3}{8}$ in. thick steel boiler plate. Difficulties were experienced in obtaining long weld runs with one type of defect, and the results do not warrant any reliable conclusions. The testing procedure is described in detail, and a review is given of previous investigations into the fatigue strength of defective welds. (13 references).—P. M. C.

Hard-surfacing with Welding. C. Stevenson. (*New Zealand Eng.*, 1955, **10**, Mar. 15, 95–96). The deposition by welding of hard wear-resistant surfaces on to a variety of engineering components is described.—P. M. C.

Better Cutting at Lower Cost. H. Bertault and J. P. Hubert. (*Rev. Soudure*, 1955, **11**, (1), 16–26). [In French]. The authors show how considerable economies in oxy-acetylene cutting can be effected by the correct handling of gas and equipment, and properly trained labour.—M. D. J. B.

Problems Arising in the Welding of Heavy Mild and Alloy Steel Parts in Modern Machinery. A. Luthy. (*Rev. Soudure*, 1955, **11**, (1), 1–15). [In French]. Examples are described of welding of heavy machine parts carried out in the works of Brown-Boveri. Details are given of the job preparation, assembly, and heat treatment prior to, during, and after welding, of the electrodes and of the weld runs.—M. D. J. B.

Relation between data on Oxygen Cutting and the Increase in Hardness in the Zones adjoining the Cut. H. R. Gunnert. (*Rev. Soudure*, 1955, **11**, (2), 68–77). [In French]. The author shows that the characteristics of the hardened zones near the cut are practically the same whether acetylene or hydrogen is used as the combustion gas. This indicates that the increase in hardness is not due to carburation due to the combustion gas. It is established that when more oxygen is used, for a given thickness of plate, the hardened zone is larger.

Auto Welding Under Pressure of Railway Rails in Germany. L. Wolff and W. Hoffman. (*Rev. Soudure*, 1955, **11**, (2), 59–62). [In French]. The authors describe a method of welding rails which consists in pressing the two carefully prepared ends together. Heat supplied by an oxy-acetylene flame accelerates recrystallization and welding.—M. D. J. B.

Temperature Distribution during Spot Welding (III), The Case of Considering the Figure and Material Constant of the Electrode. S. Ito. (*J. Mech. Lab.*, 1955, **9**, July, 136–140). The temperature distribution in plate and electrode was computed and it is shown that enlarging the radius of the electrode is the most effective means of enhancing its cooling effect.—J. G. W.

Automatic Submerged Arc Welding. G. J. M. Judd. (*Engineer Foundryman*, 1955, **20**, Sept., 48–51, 57). Heat flow and metallurgy of submerged arc-welding and the economy effected by using the process for welding a long pipe line is pointed out.—A. D. H.

Joining Metals with Adhesives. (*Mech. World*, 1955, **135**, Apr., 166–169). Brief details are given of the use of synthetic rubber and thermosetting resin adhesives.—D. H.

Double-Fillet Test for Research into Hot Cracking of Weld Metal. K. Winterton and E. G. P. Hinds. (*Brit. Welding J.*, 1955, **2**, Nov., 513–525). The double-fillet test has been developed from the T-test and is more severe than its predecessor. Investigations on the factors affecting hot cracking, using the above test, are summarized.—U. E.

Electric Welding. (*Usine Nouvelle*, 1955, **11**, Sept. 29, 55–61). Modern welding techniques are reported with particular reference to hand arc-welding, semi-automatic submerged arc-welding, automatic arc-welding, and welding in an inert atmosphere. Also mentioned are resistance welding, spot welding, and butt welding techniques.—T. E. D.

The Choice of Electrodes in the Welding of Ships. G. Zoethout. (*Trans. Inst. Eng. Ship. Scotland*, 1954–1955, **98**, 85–104). After a brief reference to the influence of design, the paper considers the different types of electrode and their development in Western Europe. Current practice in various European shipyards is reviewed.—U. E.

New High Speed Hard-facing with " Hidden Arc " Welding Cuts Equipment Repair Costs. (*Queensland Gov. Min. J.*, 1955, **56**, June, 398–400). The reclaiming of worn parts of earth-moving, crushing, and allied equipment with automatic ' hidden arc ' weld surfacing is described. The ' hidden arc ' is obtained by allowing the flux to flow on to the plate so that the arc is being completely submerged.—U. E.

Inert-Gas Shielded Tungsten Arc Welding. J. R. Baker and J. W. Ross. (*Canad. Metals*, 1955, **18**, Oct., 52–60). A description is given of recent developments in this type of welding for stainless and carbon steels using helium and argon for the inert atmosphere.—B. G. B.

Joining of Metals. J. P. Moore. (*Metal Ind.*, 1955, **87**, Sept. 30, 277–281). Some of the metallurgical problems accompanying soldering, brazing, and welding processes are discussed. The fundamental interfacial phenomena involved in each process and the mechanisms of bonding are dealt with. Structural changes and subsequent joint corrosion are also discussed.—P. M. C.

Weldability. J. F. Mercer. (*Metal Ind.*, 1955, **87**, Dec. 2, 471–472). Some of the more important factors which affect the weldability of metals are discussed. These include composition, soundness, thermal history, joint design and restraint, weld metal, preheating, and post heat-treatment.

Continuous-Cooling Transformation Characteristics related to Weldability of a 0·5% C Steel. B. J. Bradstreet and R. L.

Wilkins. (*Brit. Welding J.*, 1955, **2**, Dec., 562–566). The continuous-cooling transformation characteristics of a commercial 0·5% C have been studied by a direct-recording method, using a strain-gauge dilatometer to operate a 'function plotter' modified to draw simultaneous dilatation/temperature and time/temperature curves for a small steel specimen. The results have been related to weldability of the steel with various types of coated electrode.—U. E.

Inert-Gas Metal-Arc Process Applied to the Welding of Stainless Steel. C. L. Railton. (*Welding Metal Fab.*, 1955, **23**, Dec., 455–464). The effect of additions of oxygen to the shielding gas has been investigated and was shown to have a stabilizing effect. The effect of speed and current on the arc is discussed. Welding conditions are given for making butt and fillet welds.—U. E.

Electric Butt-welding of Grey Castings. L. Hütter and H. Neumann. (*Giesserei*, 1955, **42**, Oct. 27, 605–609). The electric butt-welding process is first described and illustrated. The influence of prior deformation on the welding and the effect of the texture are separately described and illustrated with micrographs. Finally the strength properties of the weld and the hardness changes occurring in the heat-affected zone are described.—R. J. W.

The Development and Present Position of Welding Steel No. 52. E. Folkhard. (*Schweiz. Arch. Wiss. Techn.*, 1955, **21**, June, 183–198). The development of this Mn–Si steel for welding in Germany is given and a comparison made with the welding steels of slightly different composition used in other European countries. The mechanical properties of the steel No. 52 are given and comprehensive tests of the strength of welds are described and discussed.—J. R. P.

The Influence of Shrinkage in Welded Repairs. R. Duquesne. (*Prat. Soud.*, 1955, **9**, Mar., 42–48). [In French]. The author discusses the effects of shrinkage in repair jobs and puts forward some practical suggestions for minimizing it.

The Joining of Steel-cored Aluminium Cables by an Alumino-Thermic Method. R. Mearin Domezáin. (*Cien. Tecn. Solda.*, 1955, **5**, Sept.-Oct.). [In Spanish]. Details are given of the method developed and patented by the Spanish Institute of Welding. Mechanical properties and microstructures of test welds indicate perfect results; in addition the electrical resistivity is reduced by about 5%.—P. S.

Selecting Electrodes and Welding Rods. H. Thielsch. (*Machine Design*, 1955, **27**, Sept., 187–193). Characteristics of the major electrode materials are reviewed, and the relative importance of the various classifications on the basis of their consumption are considered. Specifications have been prepared by the American Welding Society and the American Society for Testing Materials.—M. A. K.

Effect of Silicon in Submerged Arc Welds. W. Simon. (*Mat. Methods*, 1955, **42**, July, 132–134). Experiments are described in which mild-steel plate was multi-pass submerged-arc welded under granulated fluxes with different Si and Mn contents. The results of subsequent mechanical tests show that high Si content does not lower the weld-metal ductility, but that a lowering of weld metal impact values is due to relatively high Si content coupled with low Mn.—P. M. C.

Successful Build-Up by Welding of Cycle Tyre Rolls using special Austenitic Electrodes. A. Schmidt. (*Schweisstechn.*, 1955, **9**, Aug., 93). The surface of cycle tire rolls is given several layers of weld metal, the individual weld layers being hammered to prevent distortion. Stress-relief heat-treatment is carried out at 620° C after completion of the weld built-up, which has a Brinell hardness of 75–85 kg/mm². These rolls will produce 11,000 cycle tyres instead of the usual 2000 tyres.—U. E.

Alloys for Build-up Welding. H. Anders. (*Schweisstechn.*, 1955, **9**, Sept., 103–105). The problems arising when applying the built-up welding method to high alloy steels, are discussed.—U. E.

New Flux Developments In Automatic Welding. W. I. Miskae. (*Aust. Machinery Prod. Eng.*, 1955, **8**, Jan., 25, 27–29). Difficulties encountered with early fluxes in powder form for submerged-arc welding are enumerated, and the development of present agglomerated fluxes is outlined. Various applications of the process are described.—P. M. C.

Ceramic Raw Materials for the Welding Electrode Industry. W. T. DeLong and H. F. Reid, Jun. (*Amer. Ceram. Soc. Bull.*, 1955, **34**, June, 182–185). This paper summarizes the function of ceramic materials in arc welding, the specific types of materials used, the physical and chemical requirements for these materials, and finally some unsolved materials problems confronting the electrode industry.—D. L. C. P.

Surfacing with Composite Tube Rod. R. P. Culbertson. (*Welding J.*, 1955, **34**, Sept., 861–869). The tube rods used for surfacing and hard-facing consist of preblended metal powders in a continuously formed mild-steel tube. This type material may also be produced in the form of coils and cut lengths suitable for oxyacetylene welding, when coated for metallic-arc welding.—U. E.

Evaluation of Fuels and Oxidants for Welding and Associated Processes. W. B. Moen and J. Campbell. (*Welding J.*, 1955, **34**, Sept., 870–876). An improved method for the comparison and evaluation of fuel-oxidant combinations for gas heating applications is suggested.—U. E.

Shop Methods for Identifying Some Metals prior to Welding. A. L. Phillips. (*Welding J.*, 1955, **34**, Sept., 877–881). A number of tests are described for identifying properties of the material before welding.—U. E.

Nitrogen Effects in Argon-Arc Welding Atmospheres. H. C. Ludwig. (*Welding J.*, 1955, **34**, Sept., 409s–414s). The solubility of nitrogen in steel arc weld metal has been determined when using argon–nitrogen atmospheres. The results indicate greater chemical activity than that expressed by a square-root law of gas solubility with respect to nitrogen pressure in the arc-welding atmosphere. The solubility of N_2 at 1 atm was found to be in the range of 0·038–0·041 wt-%. As the metal cools to the solidus, N_2 is released to form from porosity. The porosity has been found detrimental above about 1 vol-% N_2 in the argon arc-welding atmosphere.—U. E.

An Investigation of the Fatigue Resistance of Butt Welds in a Spanish Steel of the "Lloyd" Weldable Type. Z. García Martín. (*Cien. Tecn. Solda.*, 1955, **5**, Jul.-Aug.). [In Spanish]. A study has been made of the fatigue resistance of this steel (0·08% C, 0·58% Mn) to flexural or tensile loading as affected by the introduction of butt welds made with mild steel (0·13% C, 0·39% Mn) electrodes. Graphs and results for a variety of test conditions are given. Among the conclusions it is pointed out that the fatigue resistance of an unmachined weld is only 11% less than that of the steel in its normal constructional state and that subsequent machining will give a marked improvement in fatigue resistance. Imperfect welds can cause a marked decrease, but small slag inclusions or voids do not influence the resistance of unmachined welds.—P. S.

Determination of Fatigue Strength of Welded Joints. K. Wellinger. (*Schweissen u. Schneiden*, 1955, **7**, Sept., 392–396). Fatigue tests were carried out according to DIN 50100 German Specification. The characteristic values of the various loads and the effect of shape, size, and surface quality of the test specimen on the fatigue strength were determined. The fatigue strength was greatly reduced by the presence of pores, inclusions, fissures, etc., in the weld metal and adjacent zone.

Creep Strength Tests on Welded Joints. W. Ruttmann. (*Schweissen u. Schneiden*, 1955, **7**, Sept., 397–399). The German Specification DIN 50128 for determining the creep strength of welded joints is discussed.—U. E.

Determination of Hot-Shortness of Welding Electrodes. E. Kauhausen and P. Kaesmacher. (*Schweissen u. Schneiden*, 1955, **7**, Sept., 400–404). The hot shortness of austenitic weld deposits was investigated according to the German Specification DIN 50129. Three different types of electrodes were tested and comparison was made with American weld test procedures for determining hot-shortness.—U. E.

Electric Arc Welders, with Special Reference to D.C. Machines. P. Provenzali. (*Ing. Mecc.*, 1955, **4**, Mar., 17–23). [In Italian]. A detailed examination is made of the characteristics of d.c. and a.c. electric welders. The two methods are compared both technically and economically. A special machine with current rectifiers is described and details of its performance under workshop conditions are given.—M. D. J. B.

Oxyacetylene Stress Relief in Practice. W. Liebig. (*Schweissen u. Schneiden*, 1955, **7**, Aug., 355–358). The oxyacetylene stress-relief method is discussed and experimental results are reported in a number of graphs which show that, depending on the wall thickness of welded fabrications, a 60% stress-relief can be obtained. A one-sided treatment, though, will only be effective up to a wall thickness of 23 mm. Oxyacetylene stress-relief treatment will be suitable for longitudinal and circular butt welds, and not for fillet welds or more complicated structures.—U. E.

A Simple Cracking Test for Use with Argon-Arc Welding. P. T. Houldcroft. (*Brit. Welding J.*, 1955, **2**, Oct., 471–475). The development of a simple cracking test which uses Argon-arc welding is described. Designed for research on the effect on weld cracking of material composition, the test can be used for investigating the effect of both parent metal and parent metal–filler metal combinations.—U. E.

Welding. Is it a Menace ? An Engineer's View. F. Koenigsberger. (*Manchester Assoc. Eng., Preprint*, Nov. 18, 1955). A lecture given on the possible danger of accidents and failures.—U. E.

Collapse of Large Welded Oil Storage Tanks. (*Commonwealth Eng.*, 1955, **42**, July, 473–474). The cause of failure of a 5 mill. gal. oil-storage tank constructed for Esso Petroleum Co., Ltd., was investigated. It was found that, although British Standard Specification requirements were met and weld tests were satisfactory, considerable variation in weld quality was revealed.—U. E.

Welding of Cast Iron and Steel Components in Locomotive Manufacture and Maintenance. G. Foster. (*J. Inst. Loco. Eng.*, 1955, **45**, (2), 159–203). The paper deals with the manufacture and repairs of steel and cast-iron locomotive components.—U. E.

Field Welding on Oilfield Tubular Goods. J. N. Biron and B. G. Frazier. (*J. Petroleum Techn.*, 1955, **7**, Sept., 29–32). The problems arising when welding pipelines are briefly discussed. It is shown that the tubular goods steels require a stricter welding procedure for quality welds than steels in general industrial use.—U. E.

Development and Standardization of Weld Testing Methods. K. Kautz. (*Schweissen u. Schneiden*, 1955, **7**, Sept., 374–375). A review is given of the German specifications of weld testing methods.—U. E.

Methods of Minimising Porosity of Metal Welds in Submerged Automatic Welding on Construction Sites. A. S. Fal'kevich and V. S. Volodin. (*Avtogennoe Delo*, 1951, **22**, (3), 21–23). [In Russian]. An investigation of causes of porosity of welds produced by submerged arc-welding is described. On the basis of the results obtained it is recommended that: (1) the moisture content of flux should be below 0·1%; (2) mechanical cleaning of rusty edges before welding should be discontinued; instead preheating of such edges with oxy-acetylene flame to 300–350° C and welding in two layers is recommended; (3) coating of edges of plates to be welded with an aluminium powder and kerosene mixture (25 g of Al in 1 l of kerosene) reduces very much the possibility of the appearance of porosity. This treatment is recommended for welding of $\frac{3}{32}$–$\frac{15}{64}$-in. plates of low-carbon steels having a coating of rust.—V. G.

The Tensile Test Applied to Circumferential Weld Seams of Bore-weakened Tubes. K. Kautz. (*Schweissen u. Schneiden*, 1955, **7**, Sept., 384–386). Welded tubes of 50 mm dia. and 3–5 mm wall thickness were tested using the tensile test method. Fracture was obtained in the centre of the weld seam when the weld was weakened by bore-holes. This procedure facilitated a satisfactory examination of weld quality.—U. E.

The Tensile Test for Weld Seams. J. Colbus. (*Schweissen u. Schneiden*, 1955, **7**, Sept., 376–383). The application of the German Specification DIN 50127 for notched-bar tensile test and DIN 50120 for tensile test is discussed. It is shown that fracture will not occur in the weld seam when the tensile test DIN 50120 is used. With the notched-bar tensile test, fracture will occur within the weld seam and therefore seems more suitable as a weldability test method.—U. E.

Repair Welding of Cast Iron Ingot Moulds. A. Buchholz. (*Schweissen u. Schneiden*, 1955, **7**, Aug., 335–341). Cracks in cast-iron ingot moulds are due to alternating heating and cooling of the moulds. For the repair the cracks are widened by means of the powder-cutting of oxygen-cutting methods and are then filled in by welding, using a cast-iron electrode. A metallurgical examination of the weld metal and weld adjacent zone showed a good bond and good results were obtained from tensile and bend tests.—U. E.

Inert-Gas-Shielded Tungsten-Arc Spot Welding. C. A. McClean. (*Welding J.*, 1955, **34**, July, 648–656). The variables affecting inert-gas-shielded tungsten-arc spot welds are investigated. Procedures and comparisons with resistance spot-welding and applications are discussed.—U. E.

Hand Welding 5-Chrome and 9-Chrome Pipe. W. J. Lester. (*Welding J.*, 1955, **34**, July, 641–647). A procedure that allows cooling from the pre-heat temperature to ambient conditions after a ½ h post-heat at 500° F is described. Mechanical tests in accordance with ASME code Section IX, impact tests, X-ray, microhardness surveys, and microstructure examinations were made on welded pipe lengths. It was found that the post-heat acts as a low-temperature tempering operation. Cracking of the weld is prevented when cooling to ambient temperature prior to stress relief.—U. E.

Evaluating the Iron-Powder Coated Electrode for Production Use. D. B. Howard. (*Welding J.*, 1955, **34**, Aug., 752–758). The use of iron-powder coated electrodes is discussed. All-weld-metal tension tests were carried out and it was found that for E 6012 conventional electrode and E 6024 Fe-powder coated electrode minimum yield and tensile requirements were easily met. By using interlayer rather than interpass temperature control before testing, the elongation was raised by about 30% and artificial ageing raised elongations even more.—U. E.

Mild Steel Welding in Carbon Dioxide Atmospheres. R. W. Tuthill. (*Welding Metal Fab.*, 1955, **23**, Sept., 335–338). The use of carbon dioxide gas with consumable electrodes is discussed. Welding of aluminium stainless steel, mild steel, magnesium and copper alloys has been successfully accomplished to date. A typical automatic welding unit using carbon dioxide gas is illustrated.—U. E.

Condenser Welding of Thin Products of Different Metals and Alloys. V. E. Moravskiĭ. (*Avtog. Delo*, 1953, (3), 23–26). [In Russian]. Results are presented of experiments on the welding of similar and dissimilar metals and alloys by a technique depending on the discharge of condensers through welding transformers. Typical weld microstructures are illustrated. The technique used was found suitable for spot, butt, and roller welding.—S. K.

Effect of the Type of Joint and Welding Technique on the Incidence of Hot Cracks in Welded Seams. K. V. Bagryanskiĭ. (*Avtog. Delo*, 1953, (3), 17–20). [In Russian]. After a consideration of the causes of hot-cracking in weld seams, the investigation of the roles of electrodes and techniques in tendency to this is described. Cross and **T**-joints are the most liable to hot-cracking, a beaded plate-edge the least.

Arc Welding of Sheet Clad with Steel 1Kh18N9T. N. Yu. Pal'chuk and A. N. Blinov. (*Avtog. Delo*, 1953, (3), 11–14). [In Russian]. It was found that joints of excellent mechanical and corrosion-resisting properties could be obtained in stainless-steel clad low-carbon steel sheet, using two-sided arc-welding. The sheet used in the investigation was 6, 8, or 10 mm thick, the thickness of the stainless layer being 2·0, 2·3, or 3·1 mm. Optimal conditions for welding clad sheet are summarized.—S. K.

The Use of Austenitic Electrodes for the Heterogeneous Welding of Steel. H. Granjon. (*Rev. Nickel*, 1955, **21**, July-Sept., 53–57). Different types of heterogeneous welds are illustrated. The advantages of using austenitic electrodes for these welds are discussed, and advice is given on the choice of suitable electrodes, with data on conditions under which they can be used.—T. E. D.

Electric Spot Welding Using Condenser Discharge. E. Vieilledent. (*Usine Nouvelle*, 1955, **11**, Sept. 15, 63–67). Spot-welding methods are discussed mentioning disadvantages. The circuit diagram of equipment using a condenser discharge is explained, and details of the method are given. Advantages are outlined.—T. E. D.

MACHINING AND MACHINABILITY

Abrasive Belt Polishing. L. Vorce. (*Steel*, 1955, **137**, Sept. 5, 86–89; Sept. 12, 102–104; Sept. 19, 132–134). The technique of using coated abrasive belts running over contact wheels for roughing, polishing, or finishing is described. Types of belt and contact wheel are recommended for each job. Their use in automatic production lines is mentioned.—D. L. C. P.

Sliding, Scouring and Blasting Wear by Granular Solids. K. Wellinger and H. Uetz. (*VDI-Forschungsheft*, (449), 1955, B, 1–40). The paper describes laboratory tests on the wear of various specimens by grinding materials with a range of values of hardness. Sliding contact with dry particles, rotation in water pastes (1 : 1 mixtures by volume) and blasting were used on specimens of carbon steels, alloy steels, cast irons, and other metals and non-metals. The results of the tests compare well with the effects found in practice.

Machineability of Free-cutting Steel. H. Müller. (*Stahl. u. Eisen*, 1955, **75**, Sept. 8, 1171–1176). The literature concerning the best chemical composition, melting, and processing

methods, heat-treatment, and machinability tests for free-cutting steel is reviewed and appropriate conclusions are drawn.—J. P.

Machining Tests with Steadily Increasing Cutting Speeds. A. Schepers. (*Stahl u. Eisen*, 1955, **75**, Sept. 8, 1176–1182). For rapid testing of machinability, a procedure in which the cutting speed is steadily increased has proved to be suitable. The measure of machinability is taken as that cutting speed at which the tool fails. In investigations on round turned billets of four different steels, the greatest deviations of individual values with a 17% tungsten high-speed steel tool amounted to + 2% and — 1·8%, and with a 2·25% tungsten steel tool to + 3·7% and — 4·7%. Rapid tests on Bessemer and O.H. steels showed that sulphur improved machinability more than did lead. Manganese and silicon had an unfavourable influence. Of steels which differed only in carbon content, that with 0·2% carbon turned the best. The results obtained with billets from top, middle, and bottom of ingots, as well as with billets from different casts, did not vary markedly from one to another.—J. P.

Machinability Testing—Relation Between Cutting Temperature and Tool Life for Grey Irons. E. A. Loria and D. R. Walker. (*Amer. Found. Soc., Preprint*, No. 30, 1955). The failure of cutting tools is briefly discussed and experiments carried out on unalloyed and alloyed grey cast irons to determine the relationship between tool life, tool temperature, and cutting force are described. The results show that with cast iron lower forces and temperatures give longer tool lives. The microstructure of the four irons correlated closely with tool life and temperature.—B. C. W.

Tools Last Longer, Jobs Machine Faster With Leaded Alloy Steels. H. W. McQuaid. (*Iron Age*, 1955, **176**, Aug. 18, 84–86). The production, uses, and advantages of leaded alloy or special carbon steel are noted; any grade can now be produced with uniformly dispersed particles.—D. L. C. P.

Influence of Active Coolants on Machinability of Metals and Tool Life. T. Bagassi. (*Ing. Mecc.*, 1955, **4**, Apr., 9–12; May, 21–26). [In Italian]. The author demonstrates that the machinability of metals and their alloys as well as tool life are effected by changes occurring in the atomic lattice during the cutting operation. A new concept of the function of the coolant is put forward. It is suggested that coolants to be efficient must be active, i.e. must be capable of being separated so that atoms freed from their components penetrate the metal surface deformed by the cutting process. The theoretical concepts put forward are substantiated by the practical results observed.—M. D. J. B.

Increase in the Life of High-Speed Cutters in Cutting with the Addition of Gaseous Oxygen to the Chip-Formation Zone. G. I. Yakunin. (*Stanki i Instrument*, 1955, (4), 21–22). [In Russian]. It has been shown that the life of high-speed cutters can sometimes be considerably increased by introducing gaseous oxygen into the cutting zone.—S. K.

New Developments in Grinding and Polishing Practice. W. Burkhardt. (*Metall*, 1955, **9**, June, 472–473). The spray-polishing technique is described and new tools and materials for grinding and polishing are surveyed.—J. G. W.

PROTECTIVE COATINGS

The Alloying of Tin and Iron during the Flow-brightening of Electro-tinplate. C. J. Thwaites. (*J. Iron Steel Inst.*, 1956, **183**, July, 244–253). [This issue].

PROPERTIES AND TESTS

A Study of Impact Tests and the Mechanism of Brittle Fracture. C. Crussard, R. Borione, J. Plateau, Y. Morillon, and F. Maratray. (*J. Iron Steel Inst.*, 1956, **183**, June, 146–177).

Fracture in Metals. N. F. Mott. (*J. Iron Steel Inst.*, 1956, **183**, July, 233–243). [This issue].

Ultrasonic Test Detects Enlarged Grains in Some Steel Parts. R. N. Hafemeister. (*Iron Age*, 1955, **176**, July 21, 95–97). A study is described which shows that ultrasonic testing can detect grossly coarsened grain structure in some finish-machined steel parts. The coarsening can be the result of overheating and can be the cause of service failures.—D. L. C. P.

Gamma-Radiographic Control of Welded Transmission Pipelines. C. C. Bates. (*Welding Metal Fab.*, 1955, **23**, Nov., 420–426, Dec., 465–471). A review is given of some of the methods used in the U.K. for the inspectional control of welded pipelines and relative merits of using radioactive isotopes coupled with a system of welding control. In the second part the advantages of using radioactive isotopes for non-destructive testing are discussed.—U. E.

Metallurgical Variables Influencing Properties of Heat Resistant Alloys. J. W. Freeman, C. L. Corey, and A. I. Rush. (' *Utilization of Heat Resistant Alloys* ': *Amer. Soc. Met., Symposium*, Mar. 11–12, 1954, 244–265). Published properties of alloys at high temperatures show ranges for specific alloys, these ranges being due to variations in melting, fabrication, heat-treatment, and chemical composition within specification. The author indicates how variations in melting technique (particularly deoxidation), fabrication and forming, and heat-treatment affect such properties as minimum creep rate, stress-rupture strength, etc., of high-temperature materials, giving the general principles involved with illustrations based on steel or superalloys.—D. H.

Stress Calculations for Design for Creep Conditions. P. F. Chenea. (' *Utilization of Heat Resistant Alloys* ': *Amer. Soc. Met., Symposium*, Mar. 11–12, 1954, 97–105). The author discusses the problem of forming a workable theory for stress analysis with creep and indicates what the important characteristics of such a theory would be.—D. H.

Creep and Fracture at Elevated Temperatures. N. J. Grant. (' *Utilization of Heat Resistant Alloys* ': *Amer. Soc. Met., Symposium*, Mar. 11–12, 1954, 1–28). The mathematical nature of the creep curve for pure metals is briefly discussed and some metallographic and X-ray observations of the creep phenomenon in pure Al are reviewed. Though no mathematical equations are capable of defining completely all creep curves, various methods of using creep and creep-rupture data for the purposes of extrapolation and interpolation have been suggested. The author discusses some of those theories and methods developed since 1940, in particular the rate process theory of Eyring applied to the theory of plastic deformation of metals by Kauzmann and Dushman, the structural instability method of Grant and Bucklin, the Larson–Miller method, and the techniques developed by Dorn and his co-workers.

Factors Involved in Using High Temperature Test Data for Selecting Materials and Proportioning Parts. C. L. Clark. (' *Utilization of Heat Resistant Alloys* ': *Amer. Soc. Met., Symposium*, Mar. 11–12, 1954, 29–54). Some of the factors which govern the selection of a suitable alloy and the permissible operating stress are listed. Temperature and stress, permissible deformation, and oxidation and corrosion resistance are dependent on the alloy, the expected life, uniformity of temperature, continuity of service, frequency of inspection and hazards involved are dependent on the operating conditions. Cost and availability are economic factors which must also be considered.—D. H.

Alloys and Their Properties for Elevated Temperature Service. H. C. Cross and W. F. Simmons. (' *Utilization of Heat Resistant Alloys* ': *Amer. Soc. Met., Symposium*, Mar. 11–12, 1954, 55–95). The various types of metallic material available for service at elevated temperatures are mentioned and the temperature ranges over which each type appears to be suitable and useful are indicated. The alloys discussed include Mg, Al, Cu, and Ti base ferritic steels of the C–Mo and Cr–Mo types, ferritic and austenitic stainless steels, superalloys of all types, cermets, and Mo and Mo-base. An interesting graph comparing all the alloy types discussed on the basis of stress *v.* temperature for rupture in 1000 h is included.—D. H.

Modified 18–8 Stainless for Higher Temperatures. (*Mat. Methods*, 1955, **41**, May, 117–118). To conserve supplies of superalloys it is desirable to raise the high-temperature ceiling of leaner alloys such as 18/8 steel. A brief summary is given of current work at the Cornell Aeronautical Laboratory where evaluations are being made of 100-h rupture life at 1350° and 1500° F of austenitic Cr–Ni steels modified with Ti, B, V, Zr, N, and C.—P. M. C.

Alloys for Use at High Temperatures. W. Betteridge. (*Brit. J. Appl. Phys.*, 1955, **6**, Sept., 301–306). An outline is given of the properties necessary in metals intended for use at high temperatures, especially their corrosion and creep and fracture resistances. Mention is made of the Fe–Ni–Cr system and of alloys containing no iron.—E. E. W.

Some Notes on Creep in High Temperature Alloys. W. T. Pether. (*J. Inst. Auto. Aeronaut. Eng.*, 1955, **15**, June, 80–85). A review of the problem of the failure of alloy steels at high temperatures due to creep is presented.—B. G. B.

Influence of Cold Work on Strength of Steel at Elevated

Temperatures. P. Shahinian. (*Trans. Amer. Soc. Metals*, 1955, **48**, Preprint No. 16). Stress-rupture and relaxation tests were used to determine the effect of cold work on the high-temperature properties of a quenched and tempered Cr–Mo steel. Rupture and creep strengths at 370° C were greatly enhanced by cold work. At 425° C and 480° C they were both lowered by critical amounts of cold deformation (8–15%), while beyond the critical values increased strengths were obtained. An explanation for this behaviour is given. There was no apparent influence of prior deformation on rupture strength at 540° C but creep strength decreased with increasing prior deformation. In relaxation at 480° C residual stress was lowered by an increase in cold work.—E. E. W.

The Enthalpy of a 0·12% Carbon Steel. J. R. Pattison and T. H. Lonsdale. (*J. Iron Steel Inst.*, 1956, **183**, July, 284–286). [This issue].

Studies on Mechanical Properties of 19–9 DL [Steel]. T. Hasegawa, O. Ochiai, and J. Ino. (*Tetsu to Hagane*, 1955, **41**, Oct., 1112–1118). [In Japanese]. Niobium additions to 19/9 Cr–Ni steel increased the ductility in the short-time tensile test at 700° C in the age-hardened or worked states, and in creep-rupture tests above 650° C. The effect of Nb is more pronounced at longer times or higher temperatures of test. The effects of working temperature and reduction increases were also investigated.—K. E. J.

Investigation of Boron Steel. II. S. Koshiba, K. Tanaka, and A. Inata. (*Tetsu to Hagane*, 1955, **41**, Oct., 1108–1112). [In Japanese]. The effects of B on the properties of Mn–Cr and Mn–Cr–Mo high-tensile steels were investigated. B had no effect on the Ac_1 point, but raised and lowered the Ac_3 and Ar points, respectively. Hardenability was markedly increased by 0·003% or 0·006% B, more so with the former amount. Tensile strength, yield point, and impact value were slightly increased. A satisfactory 400-kg ingot was made by addition of B in the ingot mould.—K. E. J.

Influence of Copper on Spring Steel. I. T. Kodaira and Y. Yasuda. (*Tetsu to Hagane*, 1955, **41**, Aug., 881–886). [In Japanese]. Japanese steel contains a higher proportion of Cu than most, due to raw materials, and its effect on Si–Mn spring steel was studied. Increase of Cu content led to the following: (*a*) little effect on austenite grain growth; (*b*) reduction of temperature at which cracks were generated on the eye-back portions of springs; (*c*) in the hot tensile test, a slight increase in tensile strength and yield point, but reduction in elongation and reduction-in-area; (*d*) in the hot impact test, brittleness at high temperatures; and (*e*) slight increase in resistance to deformation, measured by the dropping weight.—K. E. J.

Influence of Copper in Spring Steel. II. T. Kodaira and Y. Yasuda. (*Tetsu to Hagane*, 1955, **41**, Oct., 1098–1102). [In Japanese]. Increase in Cu content in Si–Mn spring steel at room temperature produced: (*a*) no effect on non-metallic inclusions; (*b*) increase in Jominy hardenability; (*c*) increase in tensile strength, yield point, and hardness (normalized specimens), but decrease in elongation and reduction in area; (*d*) increase in resistance to tempering, particularly at ~ 400–450° C; and (*e*) slight decrease in endurance limit of the spring leaves.—K. E. J.

The Mechanism of Boron Hardenability. Y. Imai and H. Imai. (*Sci. Rep. Res. Inst. Tohoku Univ.*, A, 1955, **7**, June, 249–261). The grain-boundary adsorptions of boron and other interstitial solute elements were metallographically examined by etching the steel with a $NaOH–NaNO_3$ aqueous solution. The observations are the basis of interpreting the hardenability in terms of B adsorption on the austenite grain surface and the effect of austenitizing temperature on the redissolution of nitrogen.—J. G. W.

Several High-nickel Alloys for Springs. (*Usine Nouvelle*, 1955, **11**, Sept. 29, 67). The chemical, physical, and mechanical characteristics of a number of recently developed high-Ni alloys are reported, with particular reference to alloys suitable for use at elevated temperatures.—T. E. D.

An Approach to the Study of the Effect of Rare-earth Additions to Steel by Use of Radioactive Tracer Techniques. C. S. Du Mont, J. E. Gates, and C. M. Henderson. (*Trans. Amer. Soc. Metals*, 1955, **48**, Preprint No. 18). Additions of radioactive mischmetal were made to an alloy of 25% Ni, 20% Cr, 3·5% Cu, and 2·5% Mo to determine the distribution of residual mischmetal constituents within the alloy. Results are given for tests on both as-cast and heat-treated specimens.

The Optimum Boron Content for Hardenability. J. C. Shyne, E. R. Morgan, and D. N. Frey. (*Trans. Amer. Soc. Metals*, 1955, **48**, Preprint No. 19). The correlation between 'effective' B content and hardenability has been established for a series of high-purity Fe–C–B alloys. A method for determination of effective B, as opposed to total B, is described.—E. E. W.

METALLOGRAPHY

"Bainitic Retained Austenite." Its "Conditioning" in En40C Steel, and Its Occurrence in Other B.S. En Steels. J. A. Cameron. (*J. Iron Steel Inst.*, 1956, **183**, July, 260–267). [This issue].

Some Experiments on the Composition of Carbides in Low-alloy Steels. J. E. Bowers. (*J. Iron Steel Inst.*, 1956, **183**, July, 268–275). [This issue].

Formation and Tempering of Martensite in 18/8 Steels. P. Bastien and J. M. B. Dedieu. (*J. Iron Steel Inst.*, 1956, **183**, July, 254–259). [This issue].

CORROSION

Corrosion Studies On A Rotary Air-Preheater. G. G. Thurlow. (*Proc. Inst. Mech. Eng.*, 1954, **168**, (20), 571–578). Tests are described which attempt to study corrosion phenomena on a model rotary air heater, under conditions similar to those experienced in industrial practice. The acid dew-point of the combustion gases varied from 240° to 320° F and maximum corrosion occurred at surface temperatures within the range 215–240° F. The results confirm previous conclusions as to the influence of the acid dew-point and of SO_3 content of the gases on corrosion.—P. M. C.

The Anodic Behaviour of Tin in Sulphuric Acid Solutions. W. Machu, A. M. Azzam, and G. M. Habashi. (*Metalloberfläche*, 1955, **9**, Apr., A58–A62; May, A73–A74). The mechanism of the two-stage passivation of tin in H_2SO_4 solution is discussed.—L. D. H.

The Cathodic Protection of Buried Pipe Lines. J. F. Bogtstra and M. Oudeman. (*Metaal Instituut TNO, Publ.*, 1955, (35), July, 39 pp.). [In Dutch]. This paper deals comprehensively with the problems connected with the corrosion of buried pipe lines attributable to electrolytic action. Numerous drawings appear indicating the various methods of cathodic protection available.—F. R. H.

Investigation on Acid-resistant High-Silicon Iron. I. Effects of Silicon and Carbon Contents on Mechanical Properties, Corrosion Resistance, and Shrinkage. H. Sawamura, O. Tajima, and K. Akamatsu. (*Mem. Fac. Eng. Kyoto Univ.*, 1955, **17**, July, 231–251). Investigations were made on irons containing 14–16% Si and 0·5–1·0% C, which, although acid-resistant, are not widely used because of brittleness, etc. Their properties are greatly influenced by the graphite content. From the viewpoint of strength and shrinkage, irons containing 14·5–15·0% Si and 0·5–0·7% C are recommended, from that of corrosion resistance, those containing 15·5–16·0% Si and 0·4–0·5% C.—K. E. J.

Contact Corrosion in Welded Ship Steel. T. Norén. (*Tek. Tidskr.*, 1955, **85**, Sept. 13, 735–738). [In Swedish]. Results are given of Swedish tests which indicate that the free boundary face between the mill scale and the actual steel is the origin of corrosion caused by galvanic cell formation, other factors such as cold-working and alkaline slag inclusions in the weld being of secondary importance. Concentrated attack along the weld seam occurs if the bare anodic (steel) surface is small in relation to the cathodic (mill scale) area. It is thus advisable to employ a method of welding in which the heat loosens the mill scale, thus increasing the anodic area.—G. G. K.

Corrosion and Protection. H. Mohler. (*Schweiz. Arch. Wiss. Techn.*, 1955, **21**, June, 204–206). A general survey of the losses caused by corrosion or its prevention is made and the annual cost to the U.S.A. is quoted and the distribution of the cost is tabulated under various headings. The problem of prevention is stated in general terms and a review of a relevant conference at Frankfurt in 1954 is briefly given.—J. R. P.

Corrosion of Instruments in Relation to Design, Manufacture, and Application. (*Trans. Soc. Instrument Techn.*, 1955, **7**, Sept.).

Scientific Basis of Corrosion. U. R. Evans. (123–124).
Design of Instruments. J. G. Walford. (124–127).
Manufacture of Instruments. B. W. Balls. (127–129).
Applications of Instruments. C. Lamond. (129–132).

The Protection of Structural Steel—Practical Experience of

Protection by Sprayed Metal Coatings—I. W. E. Ballard. (*Chem. Indust.*, 1955, Dec. 10, 1606–1611). The advantages of zinc and of aluminium coatings over normal painting methods and of metal spraying over hot galvanizing are discussed. The surface of sprayed metal is considered to form an ideal base for most paints. The economics of the spraying and painting processes are compared. Various grit-blasting processes are considered and a description is given of spraying by the wire process.—E. E. W.

The Protection of Structural Steel—Practical Experience of Protection by Sprayed Metal Coatings—II. F. A. Rivett. (*Chem. Indust.*, 1955, Dec. 10, 1612–1615). Various applications of metal spraying are described with special reference to the protection of bridges by zinc or by aluminium. Duplex coatings are mentioned.—E. E. W.

Nature of the Passive Film on Iron in Concentrated Nitric Acid. H. H. Uhlig and T. L. O'Connor. (*J. Electrochem. Soc.*, 1955, **102**, Oct., 562–572). Experimental evidence is put forward to support the view that the film responsible for passivity when iron is immersed in concentrated nitric acid is ferric acid (H_2FeO_4) or a related iron compound of higher valency. When this film decomposes it is calculated that a film of ferric oxide 40–125 Å will be formed. A mechanism is put forward for the formation of the compound and the effects of additions of urea and H_2O_2 to HNO_3 are discussed.—A. D. H.

The Electrochemical Behaviour, Including Hydrogen Overvoltage, of Iron in Acid Environments. M. Stern. (*J. Electrochem. Soc.*, 1955, **102**, Nov., 609–616). The corrosion rate of pure iron in oxygen-free acid media was studied and found to be independent of *p*H in the range 1–4 in 4% NaCl solution. The behaviour is discussed and a method is put forward for the calculation of corrosion rates from electrochemical measurements.—A. D. H.

Corrosion of Metals. S. C. Britton. (*Soc. Chem. Ind., Reports on Progress of Appl. Chem.*, 1954, **39**, 105–116). Advances in cathodic protection and in protective coatings (paints, metallic coatings, and temporary coatings) are described in some detail. Other subjects mentioned are corrosion of cast iron both at high temperatures and when buried in soils, the use of inhibitors under immersed conditions, and corrosion of ships' machinery and other metal parts inside vessels.

The Role of Nickel in the Fight Against Corrosion. M. Ballay. (*Corrosion et Anti-Corrosion*, 1955, **3**, Sept.-Oct., 213–221). Three-quarters of the world production of Ni is used for preventing corrosion. Particular reference is made to its use for this purpose in the U.S.A. The applications of pure Ni, Ni-clad steel, electrolytic Ni plating, Ni–Cu alloys and their derivatives, Ni–Cr alloys, and Fe–Ni–Cr alloys are outlined, with some performance data.—T. E. D.

Protection Against Corrosion by Hot-dip Galvanizing. P. Morisset. (*Corrosion et Anti-Corrosion*, 1955, **3**, Sept.-Oct., 238–252). Corrosion mechanisms and protection by means of coatings are outlined with particular reference to the protection of iron by Zn. The influence of various characteristics of the base metal and of the coating on corrosion protection are examined, and the behaviour of the galvanized surface in various corrosive media is studied. Applications of hot-dip galvanizing are indicated, and a list of A.S.T.M. standard specifications is given.—T. E. D.

Method for the Investigation of Contact Corrosion in Flowing Electrolytes at Elevated Temperatures. I. L. Rozenfel'd. (*Zavodskaya Laboratoriya*, 1955, **21**, (1), 60–62). [In Russian]. A method is described by which the polarity of electrodes can be determined and the corrosion behaviour in still or flowing electrolytes of metal pairs evaluated quantitatively. The metals are in the form of tubes along which the electrolyte can be made to flow. For still and flowing 3% NaCl solution the greatest corrosion among steel-containing pairs is shown by carbon steel with brass and Cu, the least by the same non-ferrous metals with stainless steel. In contact with stainless steel, Al is corroded to a considerable extent.—S. K.

Metal Coatings on Steel in Contact with Aluminium Alloys: Some Comparative Corrosion Tests. S. C. Britton and R. W. de Vere Stacpoole. (*Metallurgia*, 1955, **52**, Aug., 64–70). Steel nuts and screws coated with Zn, Cd, or Sn–Zn alloy have been exposed in contact with 5 Al alloys to suburban, industrial, and marine atmospheres, to intermittent immersion in the sea, and to salt spray in the laboratory. The results of the experiment are discussed. Considering the long term protection of the steel and Al, Sn–Zn coating is the most useful general-purpose coating of the three.—B. G. B.

Staining of Engineering Components. R. Graham. (*Corrosion Techn.*, 1955, **2**, Sept., 275–277). The author considers that at the stain age of rusting it might be possible to obtain the maximum amount of information on the rusting process. This is amplified by a consideration of the types of compounds involved, the influence of water, and the effect of surface finish. The author concludes by discussing briefly the prevention of rusting.—L. E. W.

Corrosion Research Laboratories—5. Battelle Memorial Institute, U.S.A. C. L. Peterson, F. W. Fink, and R. S. Peoples. (*Corrosion Techn.*, 1955, **2**, Sept., 270–274). A review is given of the origin and philosophy of the Battelle system of conducting research. This is followed by a brief description of some of the past and current investigations of the corrosion section of the Institute.—L. E. W.

Climatic Test Chambers. J. Grindrod. (*Corrosion Tech.*, 1955, **2**, Aug., 251–252). The author describes briefly the constructional details and applications of the climatic test chambers at the recently founded institute, near Delft, known as Klimaatkamers T.N.O.—L. E. W.

The Effect of Tempering Treatment on the Corrosion Resistance of Hardened 13% Chromium Steel. J. E. Truman. (*Corrosion Techn.*, 1955, **2**, Aug., 243–246). The author describes an investigation on the effects of various tempering treatments on three steels of differing carbon contents (0·06%, 0·23%, and 0·29%). It is concluded that (i) tempering temperatures below 400° C are safe but above this temperature the corrosion resistance is lowered, (ii) the maximum effect occurs within the range 500°–650° C, (iii) above this range the 0·23% and 0·29% C steels do not regain the resistance of the 'as-hardened' condition, and (iv) this effect is due to chromium impoverishment resulting from precipitation of chromium carbide.—L. E. W.

Corrosion of Metal Containers. R. K. Sanders. (*Corrosion Techn.*, 1955, **2**, Aug., 238–242). The author considers tinplate, 'blackplate,' and Al in their roles as structural metals for containers, outlining their corrosion characteristics under varying conditions and reviewing the present state of knowledge of metal-container corrosion.—L. E. W.

The Cathodic Protection of Ships Against Sea-Water Corrosion. L. T. Carter and J. T. Crennell. (*Shipbuilder and Marine Engine-Builder*, 1955, **62**, July, 425–432). The authors present the principles and techniques of cathodic protection in sufficient detail to indicate the advantages and limitations of the method in its application to ships' hulls. A number of circuit diagrams are included in a discussion of the application of cathodic protection to H.M. Ships in the Reserve Fleet and to active ships.—L. E. W.

External Casing Corrosion Control. J. D. Sudbury, J. E. Landers and D. A. Shock. (*J. Petroleum Techn.*, 1955, **7**, June, 92–95). The authors discuss (i) an instrument for studying external electrolytic corrosion, (ii) use of this instrument to determine the quantity of current required to prevent corrosion, and (iii) the problem of accelerated external corrosion of the oil string at the base of the surface string.

Design against Atmospheric Corrosion. H. T. Rudolf. (*Corrosion*, 1955, **11**, Aug., 35–38). Designers of metallic structures are urged to take steps to limit corrosion damage (i) by reducing the areas exposed to corrosion, enclosing with flat plates angles and braces and seal welding, (ii) by minimizing inaccessible areas so as to make surface preparation and painting possible, (iii) by substituting welding for riveting, and (iv) by attention to drainage. Examples of good and bad design are given.—J. F. S.

Kinetics of Surface Reactions of Metals. I. Iron. D. M. Sowards and N. Hackerman. (*J. Electrochem. Soc.*, 1955, **102**, June, 297–303). The reaction of iron with dilute acetic acid was studied at 34° C. The effect of variation in concentration, flow rate, temperature, and nature of reaction products on the rate of reaction are discussed.—A. D. H.

Mathematical Studies of Galvanic Corrosion. II. Coplanar Electrodes with One Electrode Infinitely Large and with Equal Polarization Parameters. J. T. Waber and M. Rosenbluth. (*J. Electrochem. Soc.*, 1955, **102**, June, 344–353). The distribution of current density over the anode and the distribution of potential within the electrolyte have been derived mathematically for the condition that one electrode is infinitely large and that Wagner's polarization parameter is equal for both electrodes. Values of the interfacial potential and the anodic current density were obtained. The results are in agreement with values determined previously.—A. D. H.

Mathematical Studies of Galvanic Corrosion. III. Semi-Infinite Coplanar Electrodes with Equal Constant Polarization Parameters. J. T. Waber. (*J. Electrochem. Soc.*, 1955, **102**, July, 420–429). A theoretical analysis is made of the influence of the proportion of anodic material and of polarization parameter on corrosion attack, assuming that anodic and cathodic polarization parameters are constant and equal. The theoretical results were compared with two experimental studies and found to be in reasonable agreement.—A. D. H.

ANALYSIS

Separate Determination of Metallic, Ferrous, and Ferric Iron When Present Together. N. I. Stognii and A. G. Kirilenko. (*Zavodskaya Laboratoriya*, 1955, **21**, (3), 294–298). [In Russian]. An investigation is described of the $CuSO_4$ method for the determination of simultaneously present metallic, ferrous, and ferric iron. The ordinary procedure gives high results for metallic iron because of simultaneous solution of iron oxides through hydrolysis of $CuSO_4$. The addition of potassium sulphate was found to suppress this hydrolysis and give results for metallic iron as accurate as those obtained by the mercuric chloride method. A method is proposed for the separation of Cu from iron oxides in ammoniacal ammonium carbonate solution in the presence of O_2.—S. K.

The Photometric Determination of Phosphorus in Steel, Mn Steel, and Cr–Ni–Mn Steel. J. Scheunemann, Jun. (*Metallurgia*, 1955, **5**, Sept., 294).

The Photometric Determination of Silicon in Steel, Mn Steel, Cr–Ni–Mn Steel, and Malleable Iron. G. Montag. (*Metallurgie*, 1955, **5**, Sept., 293).

Analytical Determination of Trace Constituents in Metal Effluents. VIII. The Colorimetric Determination of Manganese in Effluents. E. J. Serfass and R. F. Muraca. (*Plating*, 1955, **42**, Mar., 265–266). A method for the determination of 5–50 p.p.m. ammonia in the presence of 100 p.p.m. of each of 23 other cations is given. The method, involving the use of Kjeldahl distillation and Nissler's reagent, can be simplified if the concentration of each other substance is < 10 p.p.m.

The Determination of Phosphorus in Pig and Cast Iron by the Routine Nitromolybdate. W. E. Clarke. (*Brit. C.I. Res. Assoc. J. Res. Dev.*, 1955, **5**, June, 630–642). A report by the Methods of Analysis Sub-Committee of the B.C.I.R.A. on the determination of phosphorus in non-alloy pig and cast irons is presented.—B. G. B.

The Analytical Chemistry of Chromium. A. J. Blair and D. A. Pantony. (*Royal School Mines J.*, 1955, (4), 5–16). The established methods for the determination of Cr in steels are reviewed and new analytical techniques are suggested. A colorimetric method for the accurate determination in alloys over the range 0–20% is proposed and means of overcoming the presence of the interfering elements Al, V, and Co are suggested.—B. G. B.

A Rapid Method for the Determination of Magnesium in Nodular Cast Iron using E.D.T.A. H. Green. (*Brit. C.I. Res. Assoc. J. Res. Dev.*, 1955, **6**, Aug., 20–22). The principle of the method described is the titration of Mg with ethylendiaminetetra-acetic acid after separation of other interfering elements.—B. G. B.

Determination of Sulphur by Oscillographic Polarography. R. Kalvoda. (*Chem. Listy*, 1955, **49**, (11), 1631–1639). [In Czech]. The quantitative determination of sulphur, sulphates, and carbon disulphite is described. Organic solvents are used.

Colourimetric Determination of Tin Using Quercetin. K. Liška. (*Chem. Listy*, 1955, **49**, (11), 1656–1660). [In Czech]. Stannous ions were found to give a yellow coloration with quercetin in acid solutions. A rapid photometric method of determining Sn was developed on the basis of this effect. An application to Cu–Sn alloys is described in detail.—P. F.

Note on the Estimation of Traces of Cobalt in Nickel and its Iron Alloys. M. Pontet. (*Chim. Analy.*, 1955, **37**, Nov., 372–374). A full description is given of the method, in which the optical density of a toluene solution of the cobalt-β-nitroso-α-naphthol complex is measured after separation of the Fe and elimination of the Ni.—T. E. D.

Flame Photometric Determination of Manganese. W. A. Dippel and C. E. Bricker. (*Analy. Chem.*, 1955, **27**, Sept., 1484–1486). The intensity of the emission of the Mn time at 403·3 mμ is measured. The method is described together with means of eliminating interferences. Results are quoted for Mn determinations on a variety of materials including two stainless steels.—T. E. D.

Determination of Aluminium and Molybdenum in Aluminium–Molybdenum Alloys. A. Færden. (*Tidsskr. Kjemi, Bergvesen Met.*, 1955, **15**, (3), 44–45). [In Norwegian]. Details are given of an investigation of the phase conditions in Al–Mo alloys containing 1·4–1·9% Al. Alloys with up to 50% Mo were dissolved in HCl and perhydrol, and those with more than 50% Mo in HCl and HNO_3. The Mo was precipitated as sulphide and the content determined as silver molybdate. The Al content was determined in the filtrate as oxinate.

Phosphorus Determination in Grey Casting by Means of the Pulfrich Photometer. H. J. Thomann and E. Wolf. (*Giessereitechn.*, 1955, **1**, Oct., 196). The photometric method of phosphorus determination described is more economical and more rapid than the usual methods.—L. J. L.

Determination of Low Carbon Contents in Steel. Methods of Analysis Committee of B.I.S.R.A. (*J. Iron Steel Inst.*, 1956, **183**, July, 287–299). [This issue].

Potentiometric Determination of Low Carbon Contents in Steels and Ferrochrome. B. Kysil. (*Hutnické Listy*, 1955, **10**, (7), 414–417). [In Czech].

Simultaneous Determination of Sulphur and Chlorine in Coal. L. J. Edgcombe. (*Fuel*, 1955, **34**, Oct., 429–432). The use of ion-exchange resins for this type of analysis is described in detail. The S and Cl are converted into H_2SO_4 and HCl and determined volumetrically.—B. G. B.

Some Views on Sampling, with Special Reference to Reduction Materials. (*Coal and Coke*). F. L. Christiansen. (*Tidsskr. Kjemi, Bergvesen Met.*, 1955, **15**, (5), 78–84). [In Norwegian]. After stressing the economic advantages of correct sampling, the author discusses sampling methods as applied in the U.K. for small fuel, with especial reference to work by Grummell. Examples given show how heterogeneity of the material can be established and how this quality can be employed for establishing sampling methods for coal and coke based on their ash and moisture contents. Advice is given on correct sampling from conveyor belts, and wagons, together with a brief account of one sampling at Narvik by means of automatic equipment.—G. G. K.

International Methods of Analysis of Solid Fuels. J. G. King. (*Coke Gas*, 1955, **17**, Aug., 305–311). The decisions of the fourth meeting of Technical Committee 27 of the International Organization for Standardization (I.S.O.), held in Stockholm, June 6–10, 1955, are reviewed and discussed. Some details are given of methods that are, or might be, accepted as standard, with indications of difficulties involved.—T. E. D.

Rapid Gas Analyzer Using Ionization by Alpha Particles. P. F. Deisler, K. W. McHenry, and R. H. Wilhelm. (*Analy. Chem.*, 1955, **27**, Sept., 1366–1374). The analysis of a flowing or quiescent gas mixture, by means of ionization of the mixture using alpha particles from polonium in an aged radium D source, is fully described. H_2–N_2, H_2–C_2H_4, C_2H_4–C_2H_6, N_2–CO_2 and N_2–H_2–C_2H_6 have been analysed successfully. Precision in prototype apparatus was about 0·2–0·3 mole-% for binary mixtures.—T. E. D.

Flame Spectrophotometric Determination of Iron in Siliceous Materials. J. A. Dean and J. C. Burger. (*Analy. Chem.*, 1955, **27**, July, 1052–1055). Details of the technique for determining iron in materials such as alumina refractories and limestone, using a Beckman Model DU flame spectrophotometer are given. Cobalt is incorporated in the samples as an internal standard. Magnesium causes serious interference necessitating the separation of iron as hydroxide before determination.

INDUSTRIAL USES AND APPLICATIONS

Criteria in the Selection of Materials for Aircraft Gas Turbines. H. Hanink and L. Luini. ('*Utilization of Heat Resistant Alloys*': *Amer. Soc. Met., Symposium*, Mar. 11–12, 1954, 122–147). The interdependence of material properties and basic design factors is considered and the influence of specific properties on material selection for major components of a turbo-jet engine such as compressor housing, blades and discs, combustion chamber, turbine blades, and discs and bearings is illustrated.—D. H.

Fabrication of High Temperature Alloy Steel Piping Suitable for Central Station and Oil Refinery Service. R. W. Emerson. ('*Utilization of Heat Resistant Alloys*': *Amer. Soc. Met.*,

Symposium, Mar. 11–12, 1954, 184–218). A brief review is given of the development of high-temperature alloy steels for piping ranging from C–Mo steels through Cr–Mo–V steels to the highly alloyed austenitic stainless types. The metallurgical characteristics of selected materials are discussed. The welding of high-temperature piping material is discussed in some detail, together with the stress relieving of weldments and piping assemblies.—D. H.

Iron-Loss Tests on Transformers with Cores of Hot Rolled and Cold-Rolled Silicon Steel. (*Brit. Elect. Allied Ind. Res. Assoc., Tech. Rep.*, Q/T 140, 1954). Tests on three 3-phase 100-kVA transformers of identical construction show that the building losses with cold-rolled Si steels are greater than with the ordinary hot-rolled material when ordinary interleaved joints are used. In spite of this increased building loss the cold-rolled sheet give much reduced losses and magnetizing currents without resort to special core construction.—B. G. B.

HISTORICAL

Sir Henry Bessemer, 1813–1898. J. Mitchell. (*J. Iron Steel Inst.*, 1956, **183**, June, 179–188).

Sir Henry Bessemer, F.R.S. H. D. Bessemer. (*J. Iron Steel Inst.*, 1956, **183**, June, 188–189).

Sir Henry Bessemer: Inventor and Businessman. G. Delbart. (*J. Iron Steel Inst.*, 1956, **183**, June, 190–195).

Bessemer—or Steelmaking without Fuel. A. E. Chattin. (*J. Iron Steel Inst.*, 1956, **183**, June, 196–197).

The History and Practice of the Acid Bessemer Steelmaking Process in West Cumberland. F. B. Cawley and D. R. Wattleworth. (*J. Iron Steel Inst.*, 1956, **183**, June, 198–207).

MISCELLANEOUS

Problems and Progress in Manufacture and Metallurgy. H. H. Burton. (*J. Iron Steel Inst.*, 1956, **183**, June, 113–123).

BOOK NOTICES

KEATING, F. H. "*Chromium–Nickel Austenitic Steels.*" La. 8vo, pp. x + 138. Illustrated. London, 1956: Butterworths Scientific Publications, in association with Imperial Chemical Industries, Ltd. (Price 25*s.*).

It is of particular interest to have this account of the austenitic chromium–nickel steels from the user's viewpoint, based on the experience and practice of I.C.I. From the earliest large-scale application of these steels in the synthetic nitric acid plant at Billingham, the Company has been in the forefront of their development in the service of chemical engineering, and the author has, therefore, had exceptional opportunity to acquire first-hand metallurgical and service experience of his subject. He has, in fact, produced a very readable book which cannot fail to be of interest and value to all concerned with the engineering applications of such materials.

The chapters range widely, through general metallurgy, manufacturing and fabricating processes, properties and specifications, and even detailed procedure for chemical analysis. It is not surprising that in places emphasis should be uneven. This is, on balance, a gain, since it is of much greater value to have, for example, the extended consideration of sub-zero properties based on the author's direct experience, than to have more detailed accounts of more generally known or available data. For those wishing to extend their reading in specific directions the list of 58 references, while in no sense a complete bibliography, will provide very useful guidance.

It is always possible to argue over points of detail, and the present book is no exception, but the author has been at considerable pains to present a clear and fair picture, and any differences of opinion are likely to be of minor importance to the main objective. This was to provide a handbook of reference for the practical man, and has been most usefully achieved.—H. T. SHIRLEY.

"*Kempe's Engineer's Year Book for* 1955." 61st edition. 2 vols., 8vo, illustrated. Volume One, pp. xiv + 1345; Volume Two, pp. x + 1419. London, 1956; Morgan Brothers (Publishers), Ltd. (Price 75*s.* the two volumes, in case).

The additions to this publication since the 60th Edition, 1955, are mainly in the second volume and deal with civil engineering, but there are extensions to the chapters on aeronautical subjects, gas electrical engineering, and gas engineering, and to many more. The high standards of "Kempe's" are well maintained by the present contributors.—J. P. S.

KUBASCHEWSKI, O., and E. LL. EVANS. "*Metallurgical Thermochemistry.*" Second edition (completely revised and reset). 8vo, pp. xiv + 410. Illustrated. London, 1956: Pergamon Press, Ltd. (Price 55*s.*).

In form and content this book is almost identical with the original edition. As before, its main chapters are:—Theoretical Basis, Experimental Methods, Estimation of Thermochemical Data, Tables of Data, Examples of Thermochemical Treatment of Metallurgical Problems. Many sections have been improved and amplified; the part on solutions, for example, has been extended to include a section on non-regular solutions, much of the experimental entropy data there discussed having been published since the first edition was produced. The hundred pages of tables of thermal data have been brought up to date. Many of the 341 references to the tables (there are 675 in the book altogether) relate to papers published in 1955. One puzzle in the tables section is the appearance of certain names instead of numbers in the reference column—*vide* B, NbC, TaC. The section on the deoxidation of steel, which appears in the chapter dealing with typical applications of thermochemical data to metallurgical problems, has been improved by the inclusion of the manganese–oxygen equilibrium, and of newer data on the silicon–oxygen equilibrium.

Since the book endeavours to cover an extensive field in a small compass it tends to be sketchy in many places. The chapter on experimental methods, for example, is no more than an introductory survey of the more important techniques of obtaining thermochemical and equilibrium data. The limitations of some of the methods described could well be more fully stated, as in the case of the Knudsen effusion technique, and certain errors in reporting need correction.

The binding, paper, and printing are all excellent—a great improvement on the first edition, which was deplorable in these respects.

The book will be useful to all concerned in the application of thermodynamics to metallurgical problems, and this includes, or should include, physical as well as extraction metallurgists. The thermodynamics of solid alloys is now a rapidly developing subject. The theoretical sections are suitable for both honours and post-graduate students in metallurgy and the tables make it a handy reference book for those who find thermodynamics a valuable tool in solving day-to-day research and development problems.

F. D. RICHARDSON.

MCQUILLAN, A. D., and M. K. MCQUILLAN. "*Titanium.*" (Metallurgy of the Rarer Metals, No. 4). 8vo, pp. xix + 466. Illustrated. London, 1956: Butterworths Scientific Publications. (Price 56*s.*).

Since W. J. Kroll developed the magnesium reduction process in 1940 and indicated the great potentialities of titanium, research and technological development has advanced by leaps and bounds, mainly owing to American enterprise. In view of this progress it is rather surprising that no comprehensive text-book has appeared until the present volume ably compiled by the two well-known British research workers, A. D. and M. K. McQuillan. The book covers some 460 pages and is divided into 14 chapters arranged in a logical sequence covering all aspects of titanium metallurgy. Extensive references are given at the end of each section; a subject index, but no name index, appears at the end. For obvious reasons the authors have been obliged to use mainly American sources of published information. Nevertheless the work is well balanced, but the critical assessment of the information tends to be

superficial, and too much emphasis has been placed on the accuracy of physical and thermochemical data. For example, the statement that carbon cannot reduce TiO_2 "even to the limits of the oxygen bearing solid solution" (p. 6) is misleading to the general reader, who may be led to assume that the quantitative determination of oxygen by vacuum fusion in a carbon crucible would not be possible. The thermodynamic considerations for the reduction of $TiCl_4$ are incomplete (p. 11), since the free energies of the chlorides of lower valency and the reactions involved in these reductions have not been considered. An error on p. 32 in the composition of the bath used by Brenner and Senderoff, in which sodium and potassium chlorides should be substituted for the corresponding fluorides, leads to some confusion later (p. 33), where it is stated that chlorine is produced at the anode. It is not obvious why the authors suggest that fluorine should be produced at the anode, since thermodynamic considerations indicate that in a bath containing fluorides and a graphite anode carbon tetrafluoride and not fluorine would be evolved. At a sufficiently high voltage and because the concentration of fluorine ions in the bath is low, chlorine would also be evolved and form the major portion of the anodic gas.

For the temperature of transformation of titanium the authors report, among several others, A. D. McQuillan's value of $882{\cdot}5 \pm 0{\cdot}5°$ C as being the most accurate (p. 140); on p. 142 the specific heat of titanium at 15° C is quoted as $0{\cdot}1248 \pm 0{\cdot}0002$ cal/° C/g. It is doubtful whether the purity of any titanium so far produced is high enough to warrant these orders of accuracy. Again the "most probable value" of the latent heat of evaporation of titanium is given as $112{\cdot}5 \pm 0{\cdot}3$ kcal/mol (p. 142); it is questionable whether the limits of accuracy quoted are significant in measurements of this kind. The opinion expressed on p. 423 that some improvement in the scaling properties of titanium would result from alloying it with metals of lower valency is not in accordance with Wagner's theory, since such additions would increase the concentration of oxygen vacancies, and an increase in corrosion rate would be expected.

The authors have set themselves a heavy task in attempting to collate and condense the extensive amount of information available. They have performed this task admirably, and the book can be highly recommended for both the general reader and the specialist. It is impossible to cover in detail all the applications of the metal in a volume of this size, and those readers particularly interested in its uses in iron and steel manufacture will find relevant information in the recent work by G. F. Comstock.—T. H. SCHOFIELD.

MOTT, B. W. "*Micro-Indentation Hardness Testing.*" La. 8vo, pp. viii + 272. Illustrated. London, 1956: Butterworths Scientific Publications. (Price 42*s.*).

This book represents the first comprehensive treatment of all aspects of microhardness testing, and deserves special mention for clarity of presentation. The author first places hardness in perspective with other properties, and after a thorough description of the different instruments available, discusses the factors affecting the hardness figures obtained, and the controversial interpretation of the experimental data on variation of microhardness with load. This is followed by a very extensive list of applications in both metallic and non-metallic fields, and the book concludes with an examination of possible future developments. There is a bibliography of over 500 references. Apart from the inversion of Fig. 44, there appear to be remarkably few printer's errors.

The author has obviously chosen his title with some care, and implies that variation in hardness at low loads is a function of the size of the impression rather than of the load. There is a definite bias in favour of the opinion that the hardness should be constant above a critical size of impression, and then an increase at lower loads; it is perhaps a pity that this is only stated on the penultimate page.

Some striking examples of the effects of elastic deformation and orientation are noted in an interesting and important section on the indentation of non-metallic materials. Thus it is shown that under suitable conditions both diamond and graphite can be made to yield similar hardness figures. A list of the hardnesses of many important alloy constituents forms a valuable appendix.

In conclusion, this book can be confidently recommended to all users of microhardness testing equipment, and to anyone interested in the possibilities of a technique which can be applied to the study and control of plating, carburizing, diffusion, preferred orientation, alloy constitution, and the testing of an infinite variety of small parts.—A. P. MIODOWNIK.

NEWELL, W. C. (Editor). "*The Casting of Steel.*" La. 8vo, pp. viii + 599. Illustrated. London and New York, 1955: Pergamon Press. (Price 105*s.*).

This book of 599 closely printed pages brings home very forcibly to the reader the enormous accumulation of scientific and technical data on the casting of steel that has been amassed in recent years; it is divided into 13 chapters, each written by an expert on a particular subject.

The main chapter on Mould Preparation and Coremaking (a fair sized book in itself) covers the whole field from the work of the sand laboratory to the preparation of the finished mould. It is well written and copiously illustrated by excellent photographs of all the different kinds of plant used in the foundry today. This chapter is backed by two further chapters on specialized types of moulding, centrifugal and investment casting.

The remaining chapters deal with the scientific and technical information about such matters as melting, metallurgy, heat treatment, and testing, both physical and non-destructive. All of them are well written and up-to-date but space will not permit detailed criticism.

The method adopted inevitably leads to some degree of overlapping. There are, for instance, in different parts of the book two pictures and discussions of both the iron–carbon and the alumina–silica equilibrium diagrams, and a sketch, of no great interest, from Brearley's book is also given twice. Then, again, some of the theoretical information seems far removed from the practical work of the foundry; for instance, the flow of liquid in rough and smooth tubes is explained with diagrams and mathematical calculations, though it is prefaced with the remark that the information available about the flow of molten metal in channels is scanty. It must be still more scanty about molten steel cast at varying temperatures.

On the other hand, it would be an improvement if a greater variety of castings were shown stripped and complete with their runners and heads, since the study of other peoples' methods is one of the best means of self-education in the foundry.

The Editor apologizes in his preface because there are gaps which he would like to have seen filled. It must be said, however, that the gaps are very serious, since no mention at all is made of the work of the fettling shop or welding, nor is there any reference to the problem of the suppression of dust, which is one of the most difficult facing the foundryman today. In each of these fields much technical and theoretical advance has been made in recent years, and a book on steel casting is seriously incomplete without reference to them.

The criticisms must not be taken too seriously, for, taken as a whole, the book is a magnificent work of reference for the foundry manager who tries to keep up to date, and the individual chapters will be found of interest even to specialists in the various subjects.

The Editor is to be congratulated on the successful completion of what must have been an extremely difficult and onerous task.—BASIL GRAY.

ROLFE, R. T. "*Steels for the User.*" Third edition, revised and enlarged. 8vo, pp. xvi + 399. Illustrated. London, 1955: Chapman & Hall, Ltd. (Price 45*s.*).

At the present time, when so much deference is given to high-level theoretical conceptions, some of us, anyway, are apt to forget that much of the essential science of fabrication and heat-treatment operations is still largely dealt with in terms of classical metallography and mechanical testing. It is in this way that the book in question deals with carbon steels, although consideration is given to alloy steels for those applications for which carbon steels are unsuitable. The issue of a new edition is timely, especially as thirteen years have elapsed since the previous edition.

The great advantage of this book, apart from presenting a good account of the subject-matter, is that it gives clear advice, often from the author's personal experience, on the

type of steel to be employed for particular applications, and on the best condition for the steel. The matter is extremely readable, and clearly expressed in a forthright manner. There are certain points to which the reviewer objects. Thus, on p. 179 it would be preferable if the T.T.T. curve for a eutectoid steel terminated at the M_S temperature, following modern procedure. A second point is that the author indiscriminately refers to the quench product, often called nodular troostite, as bainite. Also, on p. 43 an important sub-title appears to be missing. However, these are relatively unimportant, and should not be taken to detract from the solid worth of this book, which is valuable, not only for engineers and other users of steel, but for many metallurgists as well.—A. R. BAILEY.

RUSINOFF, S. E. "*Foundry Practices.*" 8vo, pp. viii + 263. Illustrated. Chicago, U.S.A., 1955: American Technical Society; London: The Technical Press, Ltd. (Price 52*s.*).

"Foundry Practices" is essentially a textbook for the engineering degree student who requires a primer in foundry work. It will serve also as a textbook for the student taking foundry technology in a British technical college. Particularly useful in this respect will prove the set of questions which follows each chapter. It is scarcely a book for the established foundryman, although the apprentice will find it very useful.

After a general review in chapter I, the main branches of the subject follow logically in thirteen chapters of roughly similar length. Pattern-making is treated traditionally, but one fails to find reference to the newer plastic pattern-making techniques. The basic principles of mould- and core-making are well given and the methods are profusely illustrated with up-to-date photographs, diagrams, and tables. The specialist will fail to find any mention of strainer cores, whirlgate heads, atmospheric risers, and synthetic resin binders and may be annoyed at the expression "partly melted sand."

Chapters VI and VII, dealing with machine moulding and sand handling, are particularly well-written and illustrated. The detailed treatment of metal scrap and its classification is the highlight of chapter VIII, which, however, is marred by an account of the production of blast-furnace iron which is too condensed to be of any help to the uninitiated and is not required for the knowledgeable reader.

Chapter IX, devoted to metal melting furnaces, is in fair detail, so that a student reading it will be well-informed when he takes up his first job. However, it is in this section dealing with the melting of iron that the author, who is not a chemist, makes the only obvious mis-statement in the book. "The CO_2 that burns to CO at the charging door of the cupola" heralds a rather "sticky" paragraph on doubtful cupola theory. A certain weakness in foundry metallurgy is also displayed in chapter XI, but the concluding chapters on mechanical testing, metal handling, die casting, and safety in the foundry are all excellent.

The book has a satisfactory index and a bibliography containing references to 42 textbooks and 11 articles.
R. V. RILEY.

SCHUMANN, HERMANN. "*Metallographie.*" 8vo, pp. 374. Illustrated. Leipzig, 1955: Fachbuchverlag. (Price DM.30.–).

This is a poor book, and it is difficult to realize that the author has had practical experience of metallography. The first section of approximately 90 pages is devoted to a consideration of apparatus and metallographic procedure, but from the brief descriptions that are given it is improbable that one would be able successfully to carry out the every-day work of a metallographic laboratory. One reads with astonishment that the preparation of a sample for microscopic examination calls for eleven stages of polishing. Many industrial laboratories achieve the same perfection as the author with not more than five stages. The short section devoted to photomicrography is particularly inadequate and for these days misleading. The author briefly describes a method for determining the proper exposure and then gives the composition of solutions for dealing with incorrectly exposed plates. He also specifies a fixing solution based upon sodium thiosulphate, whereas for a considerable time most metallurgical laboratories have used one based upon ammonium thiosulphate which acts much more quickly.

The second section deals with simple equilibrium diagrams, and in this section one would expect to find solid solutions dealt with in considerably more detail.

The third section deals with the metallography of technical alloys, viz. of iron, copper, aluminium, magnesium, zinc, and their alloys, and white metals; it covers 180 pages, of which rather more than 100 are devoted to iron and its alloys. The author quite rightly points out the importance of the iron–carbon equilibrium diagram, but unfortunately he does not deal with its limitations. His picture of the use of carbon steel for tools might have been accurate at the beginning of this century but their use is now much more limited.

A small section is also devoted to "*Hartmetalle*" which it is said are usually classified as steels because they are made in "*Edelstahlwerken.*" He mentions three groups, viz. the easily melted cast alloys with a Brinell hardness of about 650, the less easily melted cast carbides with a hardness of 1500 to 2000, and sintered carbide with a hardness of 2000. From the text it appears that the well-known Widia hardmetal falls in the second class, i.e. is a cast alloy, and it is only when one refers to a photomicrograph (and it is a poor one) that one learns that it (Widia) is a sintered product. It is, of course, most unusual to refer to any alloys of this type as steel, and they are seldom manufactured in "*Edelstahlwerken.*"

The remaining sections are very general and a student would find them difficult to follow, whilst an experienced metallographer would learn nothing from them.

The book concludes with a subject index which leaves much to be desired, and nowhere in the text is there a reference to original sources of information, but there is an appendix of rather less than 30 references.—J. FERDINAND KAYSER.

VEREIN DEUTSCHER EISENHÜTTENLEUTE. "*Handbuch für das Eisenhütten-Laboratorium.*" Band 4. "*Schiedsanalysen.*" Herausgegeben vom Chemikerausschuss des Vereins Deutscher Eisenhüttenleute. 8vo, pp. vi + 168. Illustrated. Düsseldorf, 1955: Verlag Stahleisen m.b.H. (Price DM.22.–; members of the V.D.E. DM.19.80).

With a sub-title of Referee Analysis this is a book of wide interest, and it can be taken that the methods quoted represent the up-to-date opinion of the steelworks chemists in Germany. As such the methods can be compared with the British Standards Institution methods which have been published in numbers during the past ten years.

The preliminary section deals with solvents, solutions, reagents, and standard titrimetric substances. As an example of the latter, methods are given for the assay of ferric oxide to be used in the standardization of potassium permanganate in the method for the determination of iron.

The inspection of non-metallic materials is dealt with in detail, and methods for iron, silica, manganese, phosphorus, etc. are given, with full reference to interfering elements such as arsenic, vanadium, and copper in the method for iron where the stannous chloride reduction is used. Some groups of material, such as firebricks, fluxes, basic slag, special slag, are dealt with in particular reference to their most important elements such as citric-soluble phosphorus in basic slag. There are also in this section methods for the inspection of solid fuels, including the determination of ash, sulphur, and volatile matter.

In the next section, which presents methods for fifteen elements in steel, it is interesting to note the alternatives and their use for specific purposes, as in the case of carbon, for which three methods are set out: the strohlein method for ordinary steels, etc.; the micro-method with combustion-gravimetric finish for stainless and heat-resisting steels, and ferro-chromium; the conductimetric method for low-carbon materials. The method for phosphorus has a gravimetric finish, and once again particular reference is made to interfering elements. For sulphur only the combustion method is quoted, with no reference to a gravimetric method for steel. The methods for nitrogen are similar to accepted practice, but in the nickel method the gravimetric finish has again been adopted in preference to a volumetric one. Methods for titanium and tungsten use the absorptiometric finish, but a conventional gravimetric procedure is also given for tungsten.

The last section of the book deals comprehensively with

the analysis of the major elements in ferro-alloys and deoxidizing materials, including methods for niobium and tantalum. Methods are also included for some of the deleterious elements, such as phosphorus in ferro-manganese and tin in ferro-tungsten.

This is a well planned book with methods which are based on a wealth of experience. The detail given is exhaustive and meticulous, which is very necessary in procedures for this class of analysis. While in Great Britain more emphasis has been placed on absorptiometric methods and the volumetric finish, it is obvious that this book is planned to give the chemist a method that is considered the best for a particular element, and the book admirably fulfils its purpose.—L. E. GARDNER.

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